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INFINITE

Aerospace composites digitally sensorised from manufacturing to end-of-life

D5.1

End of life strategy

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ABSTRACT / EXECUTIVE SUMMARY	
Abstract	This deliverable presents the End-of-Life (EoL) strategy for sensorised carbon fibre reinforced composites (CFRCs) developed within the INFINITE project. The increasing use of CFRPs in aerospace and other high-performance applications, combined with the integration of glass-coated microwires for structural health monitoring, raises critical challenges regarding their sustainable disposal, recycling and reuse. The report outlines viable pathways for the reuse and recycling of these advanced composites, targeting both carbon fibres and embedded microwires. The reuse strategy focuses on assessing the state and functionality of sensorised CFRP products post-use. Given that high temperatures lead to devitrification and degradation of the soft magnetic properties essential for sensing, the study proposes criteria for evaluating whether sensorised panels can be reused in equal or less demanding applications. Analytical techniques such as hysteresis loop analysis and magnetoimpedance measurements are applied to determine sensor viability. Recycling strategies include pyrolysis and pyrolysis-oxidation treatments to recover carbon fibres and microwires from sensorised CFRP waste. Pyrolysis tests showed successful decomposition of the resin and partial recovery of carbon fibres, albeit with residues. The pyrolysis-oxidation process further improved fibre cleanliness and eliminated resin traces. Mechanical characterization of the recycled carbon fibres (rCFs), including tensile and single-fibre pull-out tests, indicated a reduction in tensile strength but retention or increase in modulus, suggesting potential for reuse in secondary applications. The embedded microwires (rMWs) were also recovered and analysed. SEM and EDX revealed partial damage to the glass coating but preserved core integrity. However, the GMI effect decreased significantly, limiting their reuse in sensing applications. Two techniques—hyperspectral imaging (HSI) and laser-induced breakdown spectroscopy (LIBS)—were evaluated for microwire identification in recycled composites. HSI proved effective, especially for surface-level detection, while LIBS lacked sufficient resolution for reliable classification. A magnet-based recovery strategy was proposed due to the magnetic nature of the microwires. An additional valorisation route is explored by converting NCF production waste into rCF non-woven mats, integrated back into new NCF products. This circular strategy supports sustainability by reintroducing recycled materials into the value chain and minimizing resource use. In conclusion, the proposed multi-path EoL strategy—encompassing reuse, advanced recycling, and reintegration into new materials—demonstrates a viable framework for managing complex CFRP waste with sensor functionalities, contributing to the broader goals of circular economy and carbon footprint reduction in advanced composites manufacturing.
Keywords	INFINITE, End-of-Life, recycling, reuse, pyrolysis, recycled carbon fibre, oxidation, HSI, LIBS

TABLE OF CONTENTS

1 INTRODUCTION

The development of novel strategies for recycling and reusing fibre reinforced plastics (FRPs) or composites with microwires (MWs) inclusion is driven by various environmental and economic factors. Recycling materials mean that wastes are processed with technically feasible and environment-friendly methods without deterioration of the mechanical or physical performance enabling their use in new production cycles. Recycling end-of-life composites is a critical challenge for the renewable energy sector.

Carbon fibre reinforced composites (CFRC) continue to play a key role in the growth and development of many modern industries. However, their proliferation has raised increasing concerns regarding the required practices and strategies to deal with these expensive engineering structures at the end of their life cycle. This has brought into focus the need to develop more sustainable and efficient recycling solutions for these products.

Similarly, glass-coated microwires contain expensive and even critical elements such as Co or B. Therefore, recycling and reusing of such materials is essentially relevant.

The aim of this task is to identify the optimum recycling process to recycle both carbon fibres and microwires and to reuse the coupons containing both carbon fibres and glass-coated microwires.

Any end-of-life strategy for the CFRC wastes should be based on the waste hierarchy stablished at European level. Figure shows the different options to manage this type of wastes trying to obtain the maximum benefit for sustainability according to the circular economy principles.

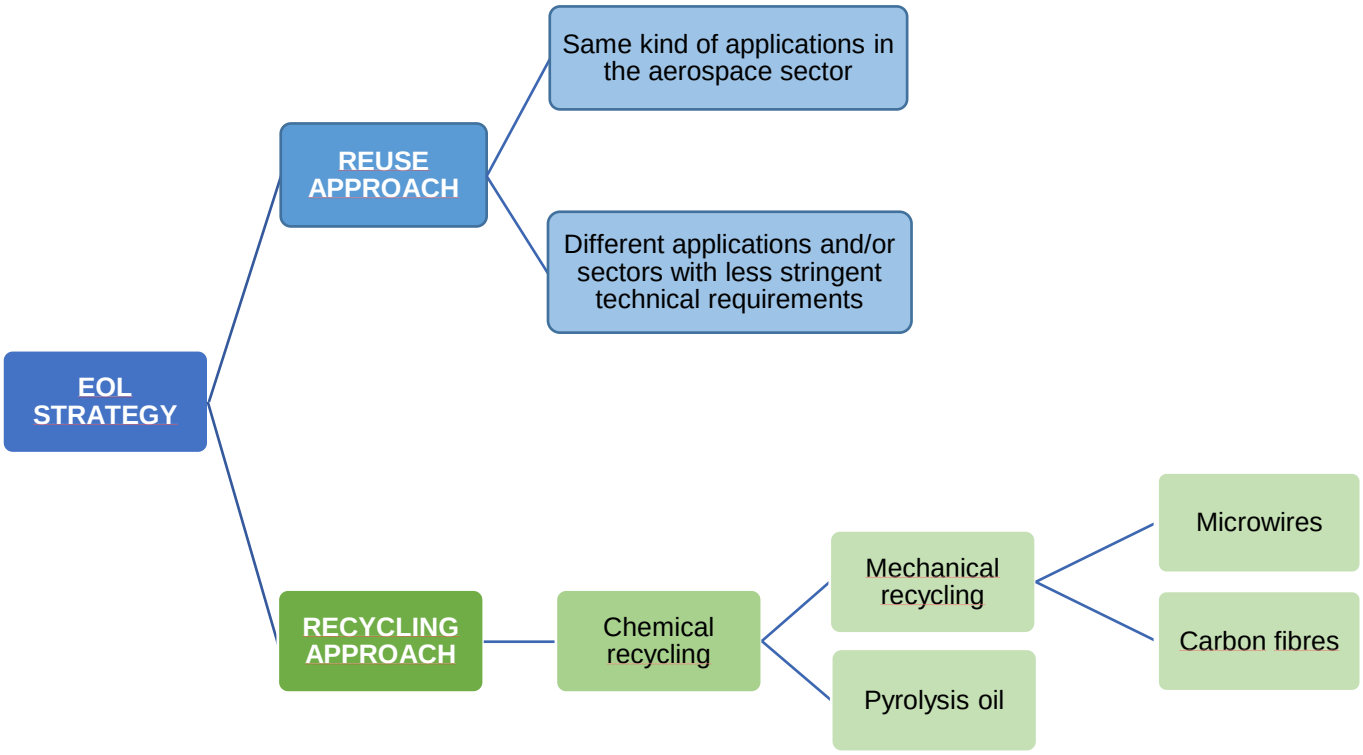


Figure 1: End-of-Life strategy for CFRC wastes

2 REUSE STRATEGY FOR CFRC WASTES (UPV/EHU)

The direct reuse and repurposing of whole parts/structures provides a significantly higher added value for materials and parts that cannot be reused in their original aviation application or are difficult to recycle.

To assess the reuse potential of any CFRC component or product the following aspects should be checked:

- **Mechanical Degradation**: Ensure that the mechanical properties (strength, stiffness) still meet the requirements for the reuse applications after years of service in aerospace conditions.
- **Thermal and Chemical Exposure**: Be cautious of applications that might involve extreme heat or chemical exposure (e.g., marine industry) since the original panels might have been exposed to similar conditions.
- **Size and Shape Restrictions**: panels reuse may be limited to applications that don't require complex shapes unless they can be cut or reshaped. Flat panels would be more accessible for reuse.

At this regard, the project has demonstrated that me presence of microwires in the CFRC panels makes it feasible checking mechanical and thermal properties of the panels, maybe not in absolute way but in properties changes, i.e. from the original panel or product, with known mechanical and thermal properties, it is possible to monitor the degradation of the panels on these properties to the point that it is possible to assess when the panel don't comply the stablished requirements during its life spam.

Therefore, it should be possible to determine the best use for these panels when they arrive to their end-of-life as a component of an aircraft, being their potential reuse applications the following:

1. *Automotive Industry*

   Non-structural parts: The panels can be reused in non-structural components like interior panels, dashboards, or door skins in high-performance cars.

   Low-load structural parts: Given their high strength and lightweight properties, they could also be applied in electric vehicle battery casings or floor panels.

2. *Marine Industry*

   Boat interiors and deck panels: The composites can be used in yacht interiors, non-structural deck parts, or cabin structures, where moderate strength and corrosion resistance are required.

3. *Renewable Energy*

   Wind turbine components: The panels could be repurposed as wind turbine blades, particularly for smaller or medium-sized turbines, due to their lightweight and stiff properties.

4. *Construction Industry*

   Facade panels: These panels can be used as exterior cladding or decorative facade panels where mechanical load requirements are less stringent, but aesthetics and durability are important.

   Reinforcement elements: Reuse in prefabricated building structures or flooring systems for lightweight, high-strength applications.

Anyway, prior to being reused in any of these potential applications, CFRC panels/components should be characterised according to international standards to check their usability in these applications.

Property	Aerospace Industry	Automotive Industry	Marine Industry
Tensile Strength	1,000 – 3,500 MPa	600 – 2,500 MPa	500 – 2,000 MPa
Compressive Strength	500 – 2,000 MPa	300 – 1,800 MPa	200 – 1,500 MPa
Flexural Strength	600 – 1,500 MPa	400 – 1,500 MPa	350 – 1,100 MPa
Shear Strength	150 – 400 MPa	100 – 300 MPa	80 – 250 MPa
Fatigue Life	10^7 to 10^8 cycles	10^6 to 10^7 cycles	10^6 to 10^7 cycles
Glass Transition Temperature (Tg)	180°C – 300°C	120°C – 220°C	110°C – 200°C
Moisture Absorption	Less than 0.5% weight gain	Less than 1% weight gain	Less than 1.5% weight gain
UV Degradation	Less than 5% degradation after 2,000 hours	Less than 10% degradation after 1,000 hours	Less than 10% degradation after 1,000 hours
Thermal Expansion (CTE)	0.5 – 3.5 × 10^{-6}/°C	0.5 – 4.0 × 10^{-6}/°C	0.5 – 4.5 × 10^{-6}/°C
Heat Resistance	Up to 250°C	Up to 200°C	Up to 180°C
Chemical Resistance	No more than 2% degradation in mechanical properties	No more than 5% degradation in mechanical properties	No more than 5% degradation in mechanical properties
Freeze-Thaw Resistance	No more than 5% degradation after 500 cycles	No more than 5% degradation after 300 cycles	No more than 5% degradation after 300 cycles
Weathering Resistance	Less than 5% degradation after 2,000 hours	Less than 10% degradation after 1,000 hours	Less than 10% degradation after 1,000 hours
Erosion Resistance	No significant reduction after 2,000 hours	No more than 10% reduction in thickness after 1,000 hours	No more than 10% reduction in thickness after 1,000 hours
Flammability	Self-extinguishing, flame spread index < 10	Self-extinguishing, flame spread index < 20	Self-extinguishing, flame spread index < 20
Creep Resistance	Less than 0.5% strain over 15 years	Less than 1% strain over 10 years	Less than 1% strain over 10 years
Aging Resistance	No more than 5% degradation after 2,000 hours	No more than 10% degradation after 1,000 hours	No more than 10% degradation after 1,000 hours
Lightning Resistance	Required, withstand up to 300 kA without damage	Not typically required	Not typically required

Property	Wind Power Industry	Construction Industry	Testing Standards
Tensile Strength	500 – 2,500 MPa	500 – 2,000 MPa	ASTM D3039, ISO 527
Compressive Strength	250 – 1,500 MPa	250 – 1,500 MPa	ASTM D3410, ISO 14126
Flexural Strength	400 – 1,200 MPa	350 – 1,000 MPa	ASTM D790, ISO 178
Shear Strength	80 – 300 MPa	60 – 200 MPa	ASTM D5379
Fatigue Life	10^6 to 10^7 cycles	10^5 to 10^6 cycles	ISO 13003, ASTM D3479
Glass Transition Temperature (Tg)	120°C – 200°C	100°C – 180°C	ASTM E1356, ISO 11357
Moisture Absorption	Less than 1% weight gain	Less than 2% weight gain	ASTM D5229, ISO 62
UV Degradation	Less than 10% degradation after 1,000 hours	Less than 10% degradation after 500 hours	ASTM G154, ISO 4892
Thermal Expansion (CTE)	0.5 – 4.5 × 10^{-6}/°C	1.0 – 5.0 × 10^{-6}/°C	ASTM E831, ISO 11359
Heat Resistance	Up to 180°C	Up to 160°C	ASTM E1461
Chemical Resistance	No more than 5% degradation in mechanical properties	No more than 10% degradation in mechanical properties	ASTM D543
Freeze-Thaw Resistance	No more than 5% degradation after 300 cycles	No more than 5% degradation after 200 cycles	ASTM C666
Weathering Resistance	Less than 10% degradation after 1,000 hours	Less than 10% degradation after 500 hours	ASTM G154, ISO 11341
Erosion Resistance	No more than 10% reduction in thickness after 1,000 hours	No significant reduction after 500 hours	ISO 22830
Flammability	Self-extinguishing, flame spread index < 20	Self-extinguishing, flame spread index < 20	UL 94, ISO 3795
Creep Resistance	Less than 1% strain over 10 years	Less than 1.5% strain over 5 years	ASTM D2990
Aging Resistance	No more than 10% degradation after 1,000 hours	No more than 10% degradation after 500 hours	ASTM D3045, ISO 4892
Lightning Resistance	Withstand 200 kA without significant damage	Not typically required	IEC 61400-24

As regarding the glass-coated amorphous microwires used as inclusions in CFRC, the crystallization of such microwires at elevated temperatures (generally above 500 °C) leads to substantial deterioration of their soft magnetic properties and mechanical properties (V. Zhukova A. C., 2001) (Churyukanova, y otros, 2014). Such crystallization depends on temperature and on time of heating.

Accordingly, after the devitrification the functionality of microwires for the present application is quite limited. Therefore, the coupons with devitrified microwires are generally not suitable for the monitoring of stresses or temperature for aircraft industry and reuse of such coupons must involve finding other applications for such coupons.

Accordingly, the plan contains:

1. Definition of the relationship between sensors signals and the composite state.
2. Definition of the criteria of the reuse considering the signals levels produced by the microwires.
3. Detection of the presence and state of microwires in the selected peace of the composite.
4. Definition of the properties that should be tested to determine whether sensors (microwires) can be reused.
5. Definition/development of the equipment necessary for the testing.
6. Definition of the industries where the coupons with devitrified microwires can be reused.

Definition of the criteria and properties of the reuse of the sensors and how they could be taken into account in the reuse process.

As regarding the relationship between sensors signals and the composite state, in the initial stage of the proposal Co-rich microwires with amorphous structure have been selected based on previous knowledge on relationship of chemical composition of microwires, their mechanical and magnetic properties.

In amorphous microwires, the magnetoelastic anisotropy, K_{me}, is the key factors that determine the character of the hysteresis loops. K_{me} is given by (A. Zhukov, 2022):

$$K_{me} = 3/2\lambda_S\sigma \qquad (1)$$

where λ_s – the magnetostriction coefficient and $\sigma = \sigma_i+\sigma_{app}$ - total stresses, σ_{app} - applied stresses.

The λ_s sign and value of amorphous materials is primary influenced by the chemical composition (A. Zhukov, 2022).

Vanishing λ_s values can be obtained in Co-rich microwires (A. Zhukov, 2022).

The core of the technology of smart composites with magnetic wire inclusions is the tunability of the effective permittivity at the GHz range due to such ferromagnetic microwire inclusions presenting the high frequency impedance sensitive to tensile stress, temperature and magnetic field (D. Makhnovskiy, 2008) (L. Panina, 2011). The principal advantage of this technology is that proposed free space microwave spectroscopy allows remote monitoring of external stimuli, like stress, magnetic field or temperature.

For achievement of high Giant Magnetoimpedance, GMI, effect is essential to have soft magnetic behaviour with high magnetic permeability. Such conditions can be obtained in Co-rich amorphous microwires with vanishing λs - values.

Generally, upon devitrification of Co-rich amorphous microwires a degradation of both magnetic softness and mechanical properties take place (V. Zhukova A. C., 2001).

Therefore, after heating at high enough temperatures or times, it is expected that the coupons with magnetic wire inclusions will be unsuitable for the applications involving contactless stresses or temperature monitoring.

In the laboratory of the UPV/EHU we performed a comparison of the hysteresis loops of $Co_{72}Fe_4B_{13}Si_{11}$ microwires (d=40 μm, D=45 μm) selected for the preparation of all the coupons based on their magnetic softness and high GMI effect.

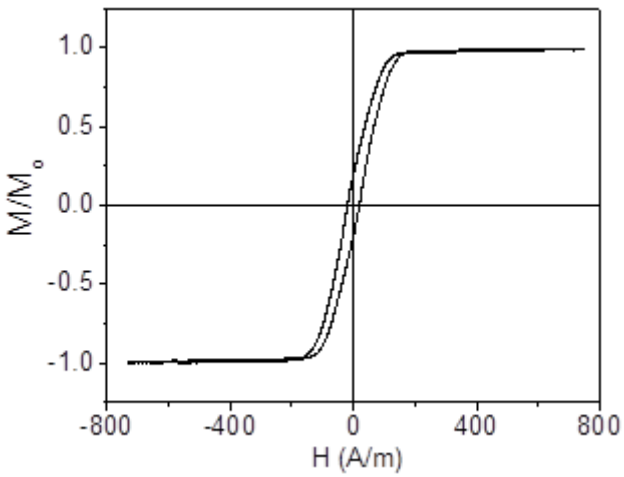


Figure 2: Hysteresis loops as-prepared $Co_{72}Fe_4B_{13}Si_{11}$ microwires (d=40 μm, D=45 μm)

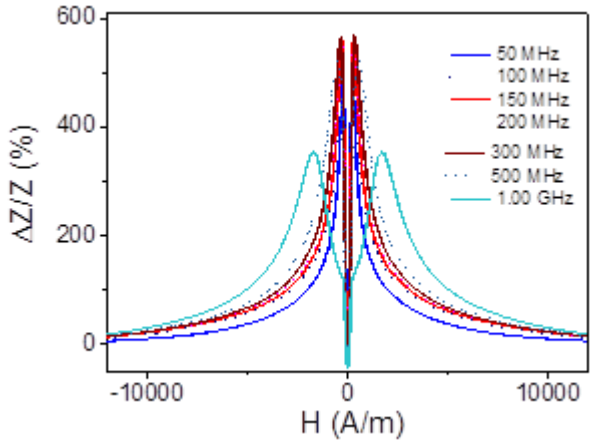


Figure 3: ΔZ/Z(H) dependencies measured in as-prepared $Co_{72}Fe_4B_{13}Si_{11}$ microwires (d=40 μm, D=45 μm)

As shown in *Figure 2*, selected $Co_{72}Fe_4B_{13}Si_{11}$ microwires present good magnetic softness (H_k ≈130 A/m, H_c ≈20 A/m) and even most relevant, these microwires present higher GMI effect (see *Figure 3*) with maximum GMI ratio up to 600% (among the highest GMI ratio reported up to now).

After the crystallization of this microwire substantial deterioration of magnetic properties takes place, as shown in *Figure 4*.a. The coercivity, H_c, of the $Co_{72}Fe_4B_{13}Si_{11}$ microwire after crystallization is about 20,000 A/m. Such Hc – value is 1,000 time higher that of amorphous microwire of the same composition (H_c ≈20 A/m). For comparison, the hysteresis loops of amorphous $Co_{72}Fe_4B_{13}Si_{11}$ microwire and the same microwire after crystallization are provided in *Figure 4*.b.

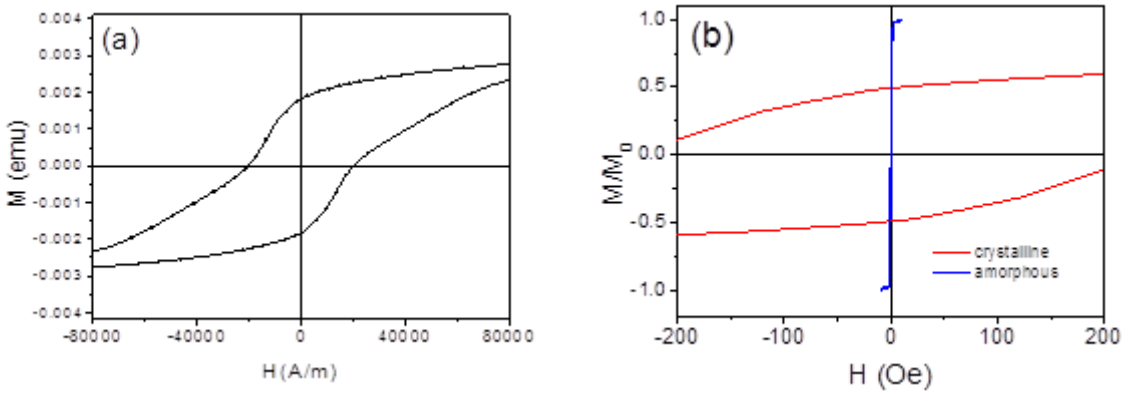


Figure 4: Hysteresis loop of crystalline $Co_{72}Fe_4B_{13}Si_{11}$ microwire (a) and a comparison of the hysteresis loops of amorphous and crystalline $Co_{72}Fe_4B_{13}Si_{11}$ microwires (b)

In proposed method of detection of amorphous microwires inclusions in carbon fibre composites a low modulating magnetic field with amplitude, H_o, of about 800 A/m is used (V. Zhukova M. I.-L.-G.-E., 2024). Use of such low modulating magnetic field allows to separate the signal originated by the magnetic wire inclusions from the microwave signal produced by conductive Carbon fibres. As evidenced from *Figure 4*, such modulating field with low H_o –value cannot produce any substantial change in the magnetization of crystalline microwires, while a complete remagnetization of amorphous $Co_{72}Fe_4B_{13}Si_{11}$ microwire is produced by such modulating field with $H_o \approx 800$ A/m.

3 RECYCLING STRATEGY FOR CFRC WASTES

3.1 CHEMICAL RECYCLING: PYROLYSIS (GAIKER)

A study of the recycling of CFRCs through the pyrolysis process has been carried out. The study aims to recover carbon fibre and magnetic microwires. For this purpose, pyrolysis tests have been carried out with a sample of sensorised composite in order to fine-tune the process. Then, the pyrolysis process was studied under the optimum conditions. The results suggested that some resin remained on the fibres. To improve the cleanliness of the fibre surface, pyrolysis-oxidative tests were conducted.

3.1.1 METHODOLOGY

Pyrolysis is an interesting alternative to recycle sensorised composite and to recover valuable materials while preserving their physical and chemical properties. During pyrolysis, composite waste is heated in inert atmosphere and organic compounds are broken down into smaller molecules. The process takes place over a broad temperature range, between 300 and 700 °C, though the exact temperature depends on several factors, including the type of waste, residence time in the reactor, heating rate, use of catalysts, and the desired end product. The resulting materials are divided into three main fractions: gases, liquids, and solids, with their proportions varying based on the original material.

The main goal of sensorised CFRC waste pyrolysis is to separate three key components: carbon fibre, resin, and microwires. Carbon fibre and microwires complete the solid fraction. Meanwhile, the resin decomposes into condensable gases, which are collected as pyrolysis oil, the liquid product of the process. Non-condensable gases are not collected but are analysed by micro-GC technology to determine their composition.

Thermal cracking tests were carried out under nitrogen atmosphere in a reactor manufactured from SS316 stainless steel designed to operate at atmospheric pressure, or alternatively a maximum of 2 bar and a maximum temperature of 750 °C. The maximum capacity of the reactor is 5 L and it has a cylindrical shape with an ellipsoidal bottom (Figure 5).

Figure 5. Pyrolysis reactor

The product collection system consists of 2 shell tube condensers. The first condenser could operate temperature up to 120°C. This condenser is connected to a liquid-gas separator with a capacity of 3.5 L, where there is a discharge valve for liquids and a vent outlet for gases. This vent outlet is connected to the second condenser, which, in turn, is cooled by a chiller that can operate at temperatures down to -10 °C. This condenser is also connected to a liquid-gas separator with a capacity of 1L to obtain the condensed gases.

To verify temperature range to run pyrolysis reaction, thermogravimetric analysis (TGA) was used. The thermogravimetric analysis was carried out with Mettler Toledo equipment, model TGA/DSC1 (Figure 6). The analysis of the samples, was carried out in nitrogen atmosphere, with a heating rate of 20 °C/min, from room temperature (25 °C) up to 800 °C.

Figure 6. Mettler Toledo equipment for TGA

By this analysis, the mass degradation of each sample was evaluated, and its derivative was used to obtain the temperature of maximum degradation rate.

Carbon fibres have been analysed using Scanning Electron Microscopy analysis (SEM). The equipment (Figure 7) used consists of a ZEISS scanning electron microscope (model EVO50) with a coupled OXFORD X-ray detector. The microscope has a secondary electron detector (SE) and a backscattered electron detector (BSD).

Figure 7. SEM equipment

3.1.2 TESTS

Tests

The pyrolysis process has been studied as a method for recovering microwires and recycled carbon fibres. For this reason, several tests have been carried out, setting the operating parameters to determine the optimal configuration of the process.

The sensorised composites samples provided by UPV/EHU (Figure 8) are:

- Sample F, marked as #23116, was a sample manufactured by IDEKO with a thickness of 4 mm, and MWs of 4 mm each. This sample has CF layers at +0°/90° angles, with MWs placed between the CF layers and,
- Sample G, identified as #23113, was also manufactured by IDEKO, with a thickness of 8.3 mm and MWs of 3 mm each. In this case, the CF layers are oriented at +45°/-45° angles, with MWs positioned between the CF layers.

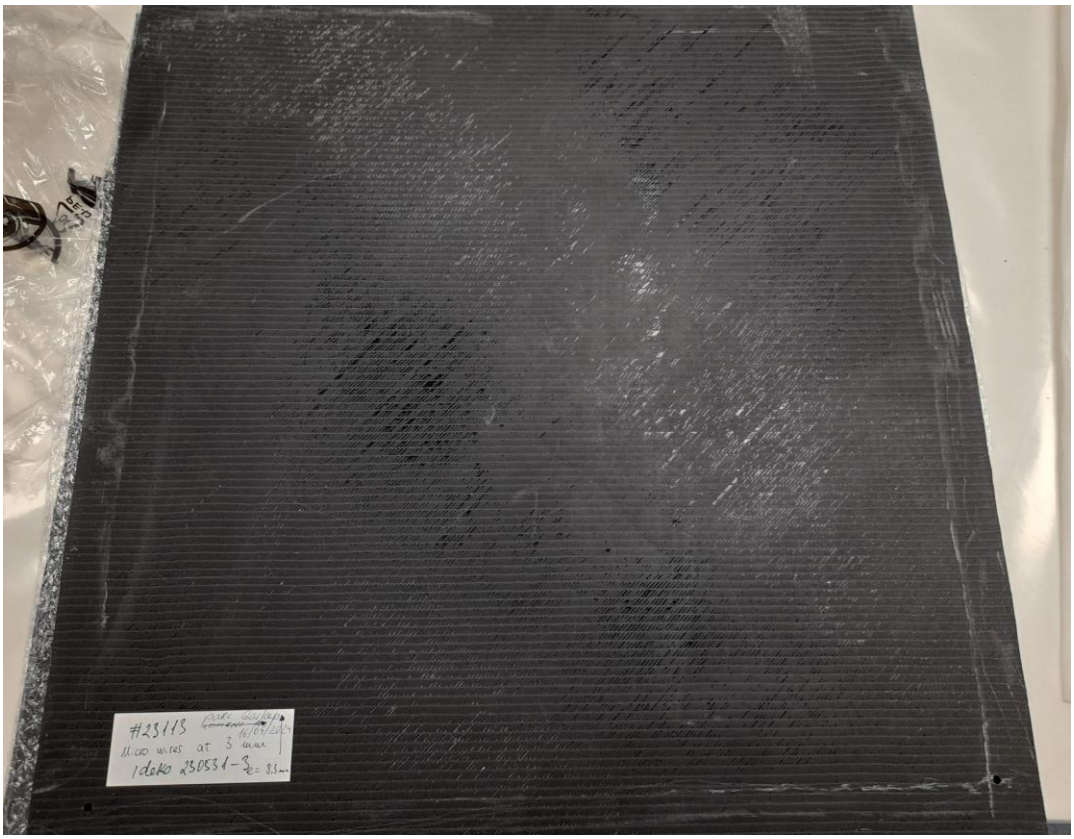


Figure 8. Sensorised CFRC waste sample

By TGA analysis, the mass degradation of each sample was evaluated, and its derivative was used to obtain the temperature of maximum degradation rate. The temperature of maximum degradation rate for sample F was 355 °C (Figure 9) and for sample G was 385 °C (Figure 10).

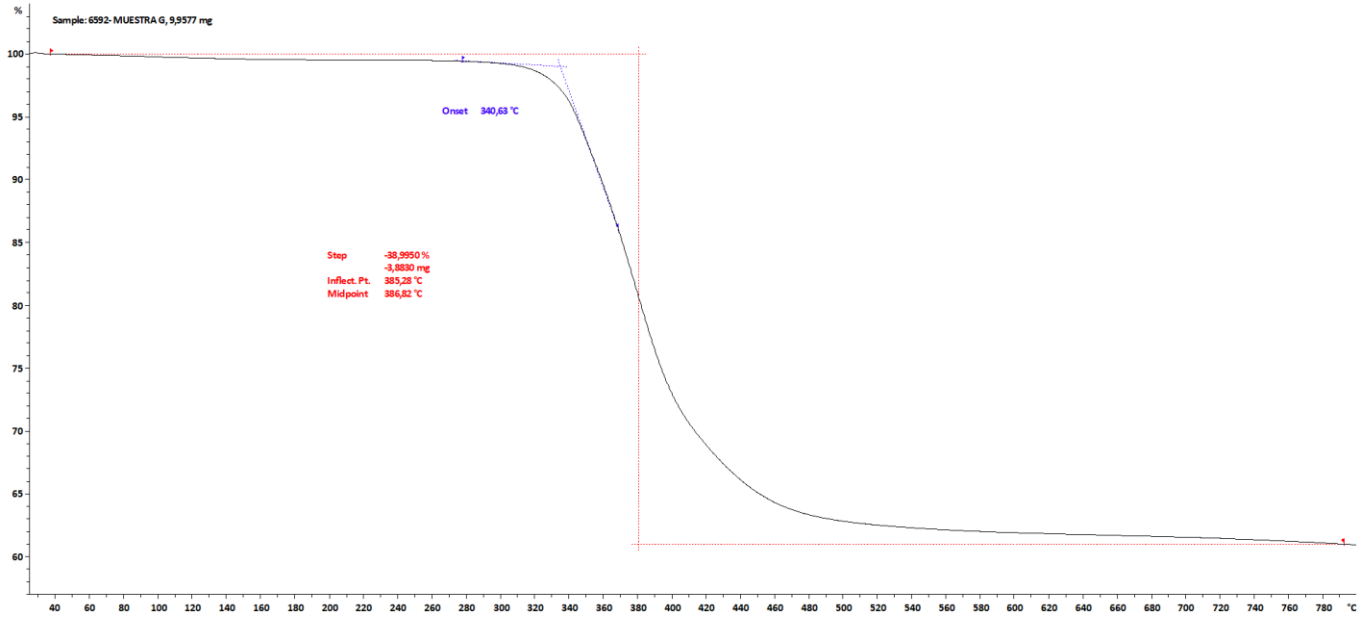


Figure 9: Sample F TGA

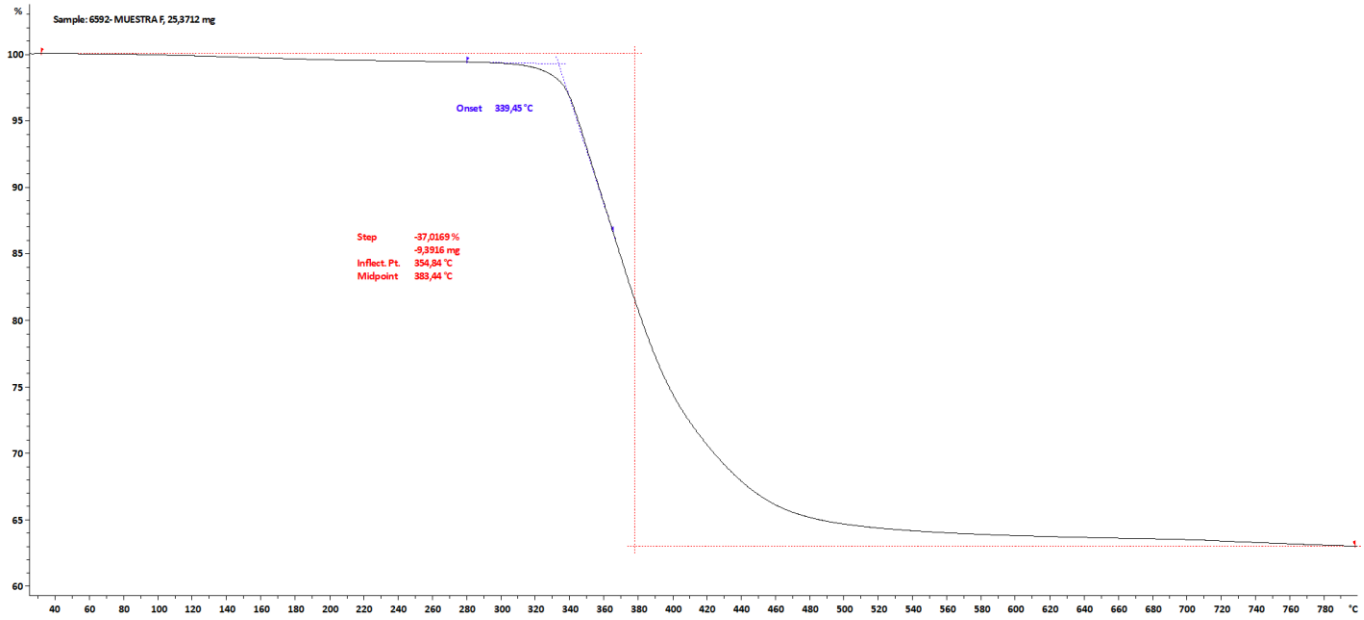


Figure 10. Sample G TGA

Sample conditioning for pyrolysis tests

To carry out the first pyrolysis tests, sample F was selected due to its lower thickness and lower pyrolysis temperature. Before pyrolysis, the sample must be conditioned to reduce its size to the size of the pyrolysis reactor chamber. The composite received was 500x500 mm and the size has been reduced to 100x100 mm to feed the sample into the reactor (Figure 11).

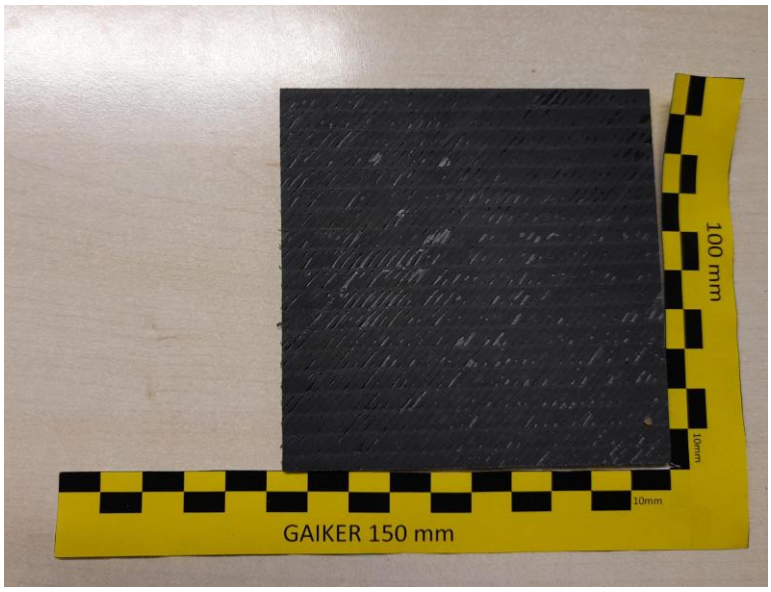


Figure 11. Samples pre-treated for pyrolysis study

Preliminary pyrolysis test

Pyrolysis is a thermal process that involves the decomposition of polymers by applying high temperatures in an inert atmosphere, i.e., without the presence of oxygen. During this procedure, the materials are heated to decompose the polymer, generating by-products such as pyrolysis oil, char (a carbon-rich solid residue), and gases. The aim of pyrolysis in this context is to depolymerize the resin to release and recover carbon fibres and microwires, allowing for their separation and subsequent recycling, which contributes to a more sustainable management of composite materials.

The thermal cracking test was conducted under a nitrogen (N_2) atmosphere at two different temperatures: 355 °C and 390 °C. The purpose of selecting these temperatures was to ensure that the material would ensure the pyrolysis process. The heating rate applied during the test was 6.7 °C/min, allowing for controlled thermal decomposition and accurate monitoring of the process.

Table 1 shows the results of the yields of the products obtained in the pyrolysis tests. The quantities of solids and liquids after pyrolysis were weighed and their yields were calculated according to the Equation (2), where y is the fraction (liquid/solid) to be calculated. The gas yield was calculated by difference. Additionally, the data of the liquid fractions recovered in the different condensers were included.

$$Yield\ y\ (\%) = \frac{mass\ y\ (g)}{mass\ waste\ feed\ (g)} \times 100 \qquad (2)$$

Table 1: Pyrolysis products yields

Essay Code	Liquid yield	Solid yield	Gas yield
Sample_F_1	10.64 %	70.93 %	18.43 %
Sample_F_2	2.22 %	71.46 %	26.32 %

Figure 12 shows the r-CF after pyrolysis, whilst Figure 13 presents the SEM analysis of r-CF after pyrolysis test.

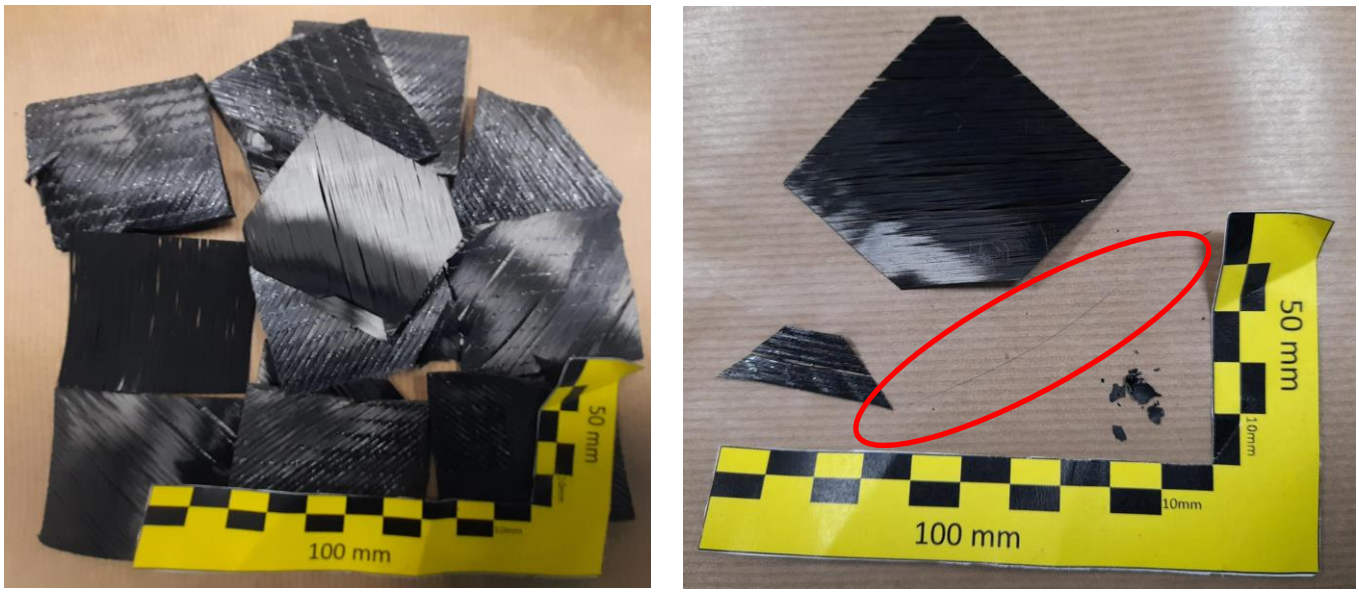


Figure 12: r-CF after pyrolysis test

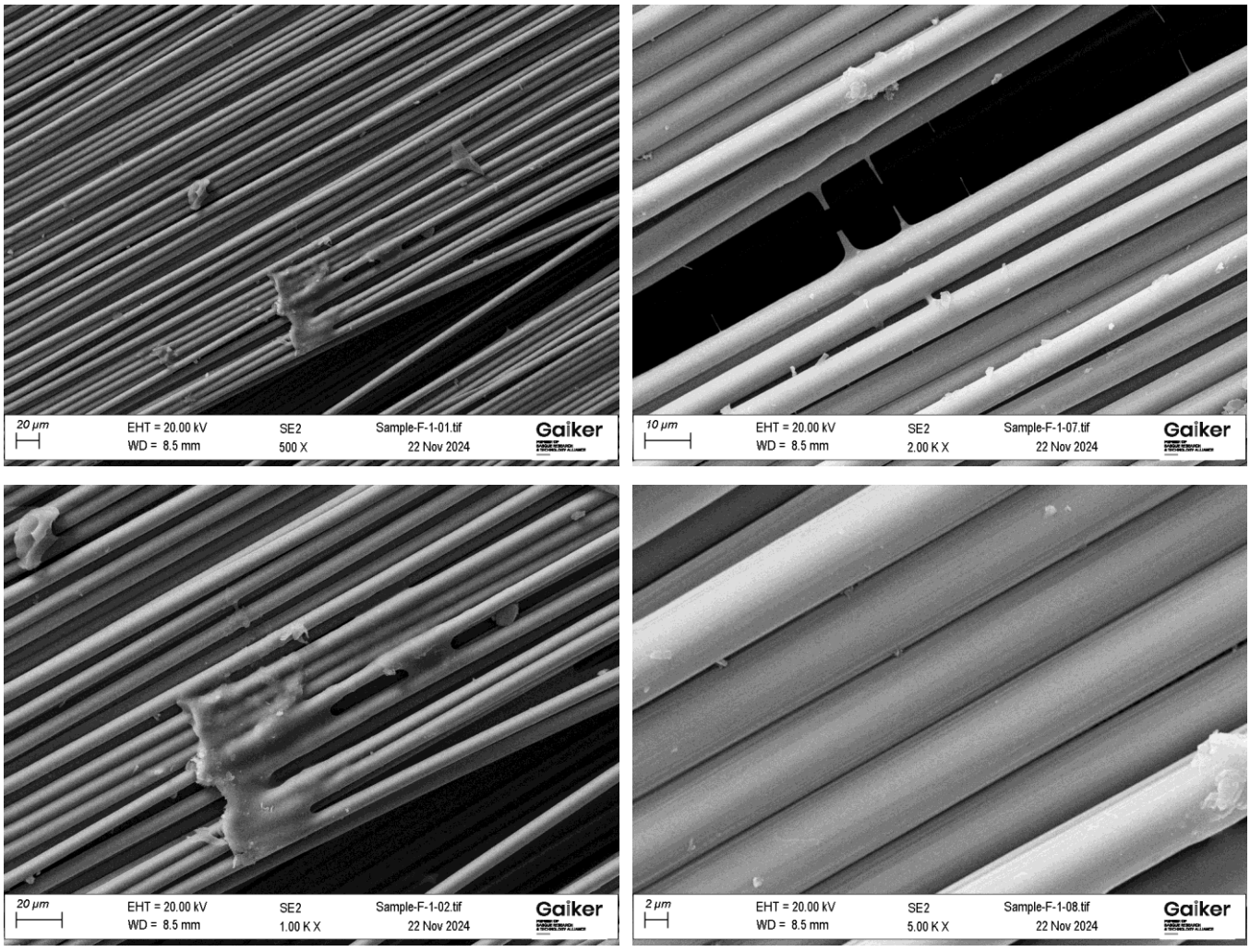


Figure 13: r-CF SEM

In the analysis it is observed how the carbon fibre has impregnated residue. This could be the resin that has not yet been pyrolyzed or the char formed in the pyrolysis process. Therefore, a new strategy is proposed to eliminate the impregnated material. Calcination has been selected as the method to remove the remaining impurities, following the guidelines of ASTM D3172 Procedure H. This approach aims to thermally decompose any residual organic matter at 550 °C in an inert atmosphere, ensuring a cleaner fibre surface. Calcination tests have shown that the loss of mass was only 5 %, the rest being carbon fibres impregnated with char. To ensure these results a SEM and a TGA analysis have been made (Figure 14 and Figure 15).

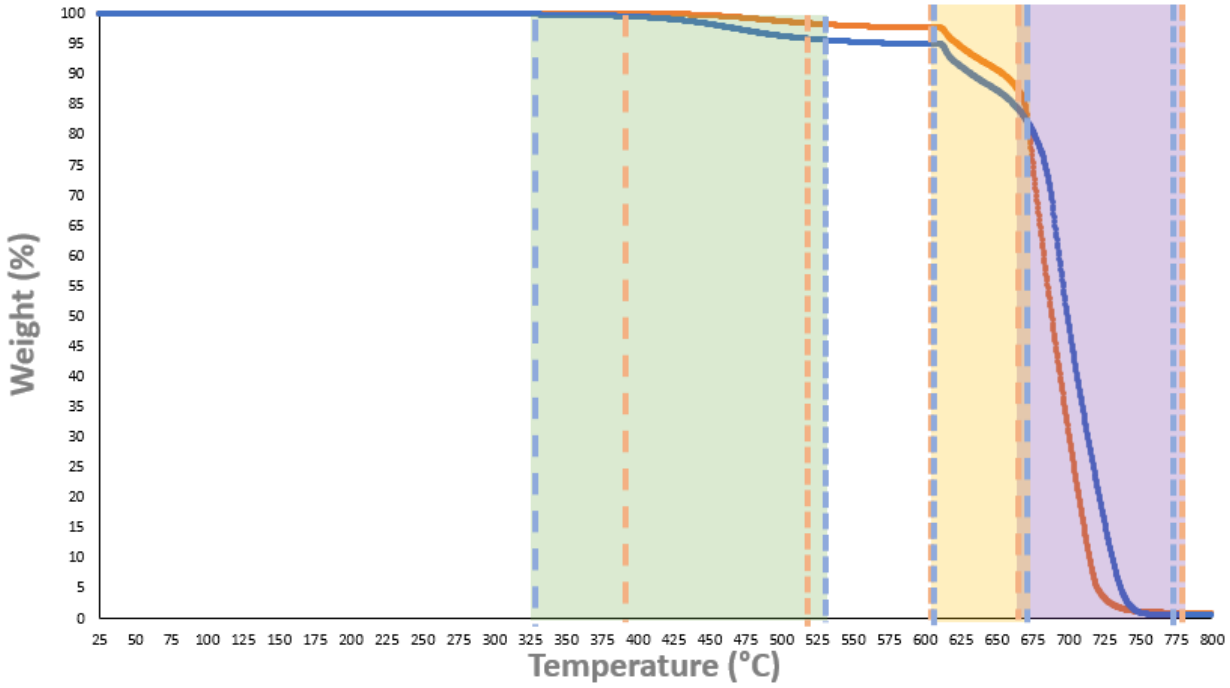
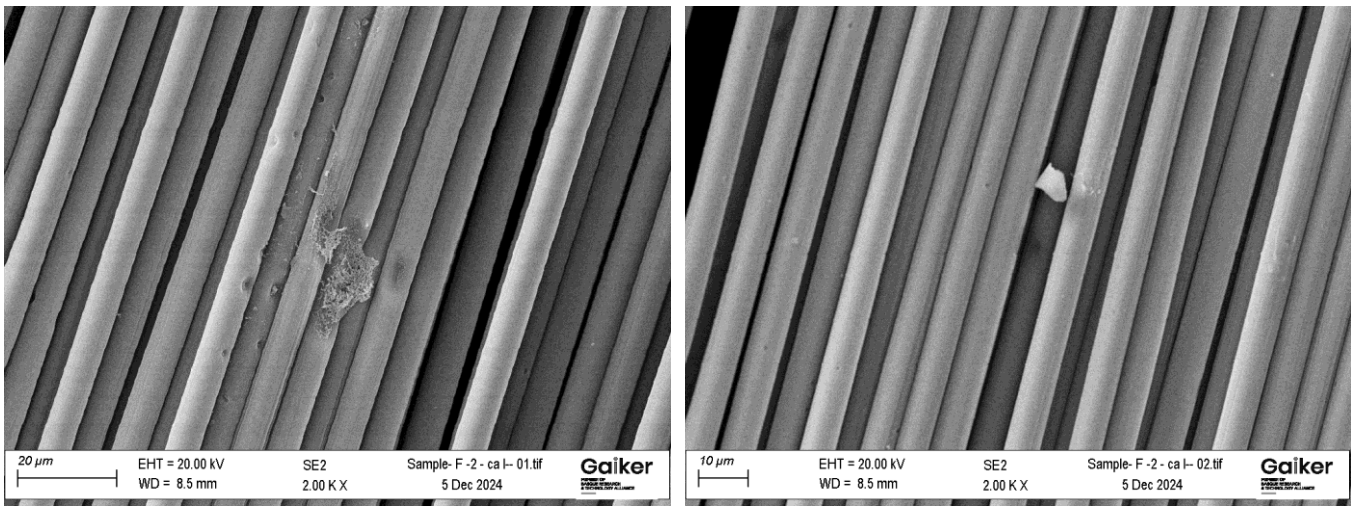


Figure 14. Calcined r-CF TGA



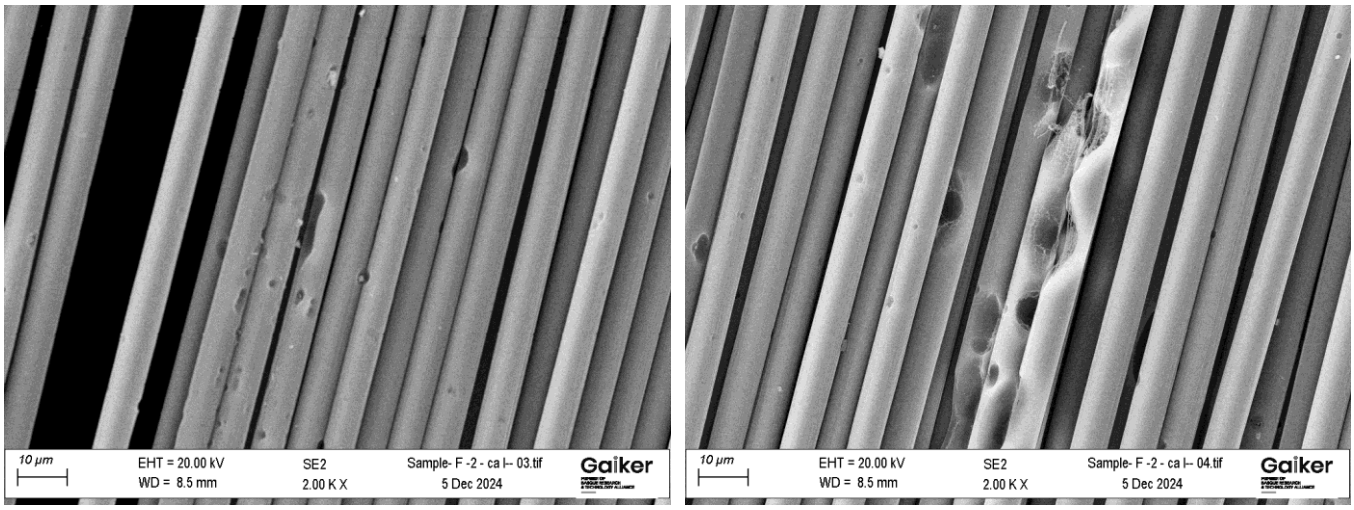


Figure 15. Calcined r-CF SEM

As shown in the analysis, the impregnated residue has been removed from the surface. Nevertheless, some fibres appear to have been damaged during the calcination process, although the majority remain intact.

In view of the results of calcined r-CF, an oxidative stage is added to remove the organic waste and the char formed during pyrolysis.

Pyrolysis-oxidative test

Several experiments were conducted to fine-tune the process and perform the oxidative stage. Table 2 lists the experiments performed. The pyrolysis process was carried out under two different atmospheric conditions: inert and oxidative. The reaction temperature was set at 390 °C. Once the pyrolysis temperature was reached, it was maintained for 120 min. In the oxidative atmosphere stage, air was introduced to promote the oxidation of residual materials. The reaction temperature was increased to 600 °C and it was maintained for a specific period.

Table 2. Experiments performed for the oxidative process tuning

	Operating parameters inert atmosphere				Operating parameters oxidative atmosphere			
TEST	Reaction Temperature (°C)	Heating rate (°C/min)	Isotherm time (min) with N_2	N_2 flow (mL/min)	Reaction Temperature (°C)	Heating rate (°C/min)	Oxidation time (min)	Air flow (ml/min)
Infinite_01_ant_F	390	6.17	120	500	600	10.5	60	1000
Infinite_03_ant_F	390	6.17	120	500	600	11.67	120	1000
Infinite_04_ant_F	390	6.17	120	500	600	10.5	90	1000

The experiments showed that with a shorter duration (60 min), the fibres were rigid and brittle, with visible char remaining. After 90 min, the fibres were no longer rigid, and no char was observed. However, at 120 min, the fibres were damaged. *Figure 16* shows the results of the TGA analysis of the products obtained after test 01 and 04, where curve 04 decreases at an earlier temperature and more abruptly than curve 01, This indicates that the char has been removed from the fibres. Additionally, in both oxidative-pyrolysis test, test 01 and 04, the resin impregned in the test sample_F_1 has been removed. For these reasons, the optimum conditions for the pyrolysis-oxidative test were set at an oxidation time of 90 min, in order to obtain high quality r-CF.

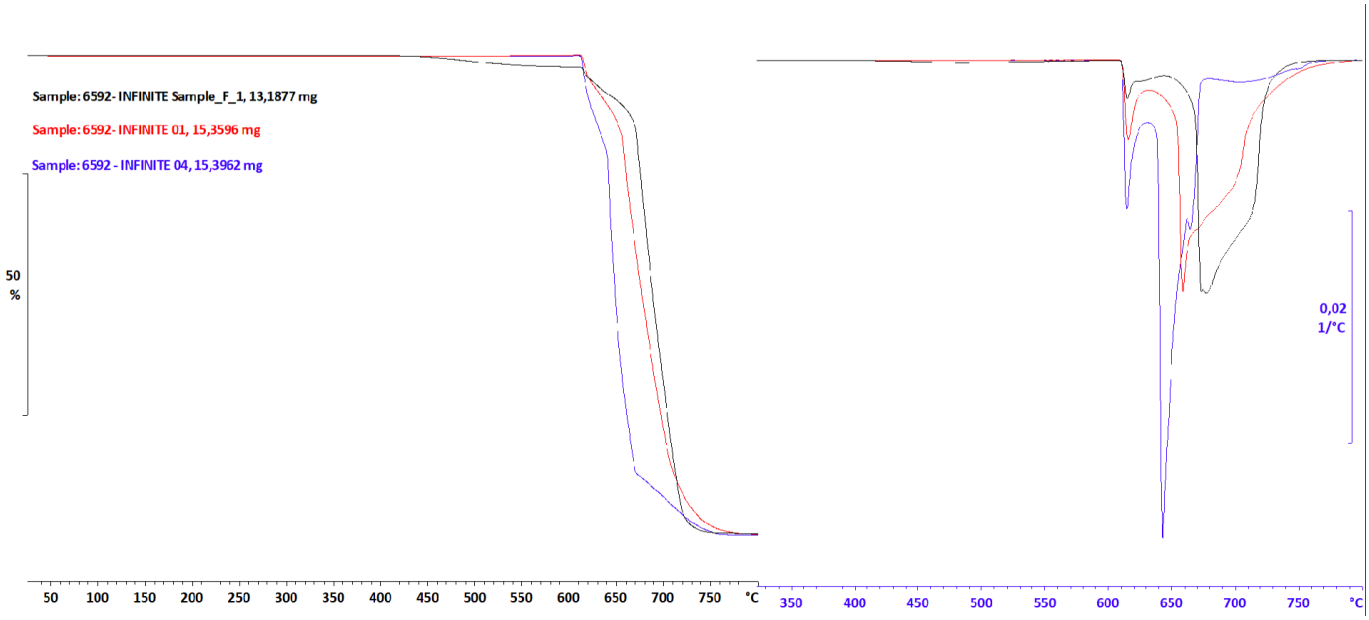


Figure 16: Oxidative process tunning – TGA

Taking in to account the previous results the final sensorised composite was pyrolyzed under the same conditions as Infinitive_04_ant_F. Table 3 shows the conditions under which pyrolysis-oxidative process was performed.

Table 3. Pyrolysis-oxidative optimum conditions

	Operating parameters inert atmosphere				Operating parameters oxidative atmosphere			
TEST	Reaction Temperature (°C)	Heating rate (°C/min)	Isotherm time (min) with N_2	N_2 flow (mL/min)	Reaction Temperature (°C)	Heating rate (°C/min)	Oxidation time (min)	Air flow (mL/min)
Infinite_PO	390	6.17	120	500	600	10,5	90	1000

The Table 4 shows the results of the yields of the products obtained in the pyrolysis-oxidative tests.

Table 4. Pyrolysis-oxidative products yields

Essay Code	Liquid yield	Solid yield	Gas yield
INFINITE_PO1	22.19 %	72.69 %	5.13 %
INFINITE_PO2	16.83 %	71.89 %	11.28 %
INFINITE_PO3	17.06 %	71.50 %	11.44 %

The product obtained can be seen in the figure below Figure 17. This product has also been TGA and SEM to characterize it.

Figure 17: r-CF (P-O) TGA

The results of the TGA tests, shown in Figure 18, reflect that the r-CF obtained didn't keep any trace of resin.

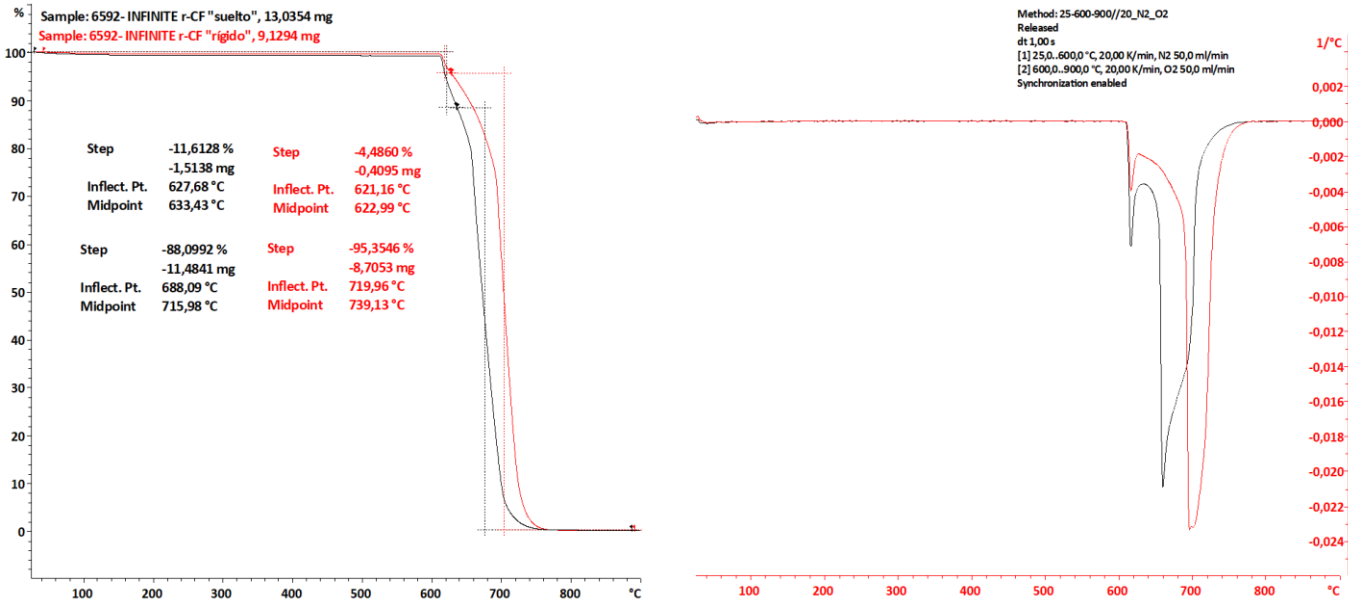


Figure 18. r-CF (P-O) – rigid rCF on the left and loose rCF on the right

After the oxidative pyrolysis, two different types of r-CF were obtained, as shown in Figure 19. In the left column, images of a rigid r-CF obtained are shown, while on the right column, a loose r-CF is shown. The rigid r-CF obtained was very fragile and apparently, fibres are joint with some residue gluing fibres. This could be related with the way the CFRP waste was loaded into the reactor, with no space between coupons which could difficult the flow of oxygen during the oxidation phase of the recycling process, and therefore making difficult the destruction of these potential organic residues. On the other hand, the loose r-CF shows, apparently, a good aspect, and as can be seen, fibres look quite clean, with no residues and few damaged.

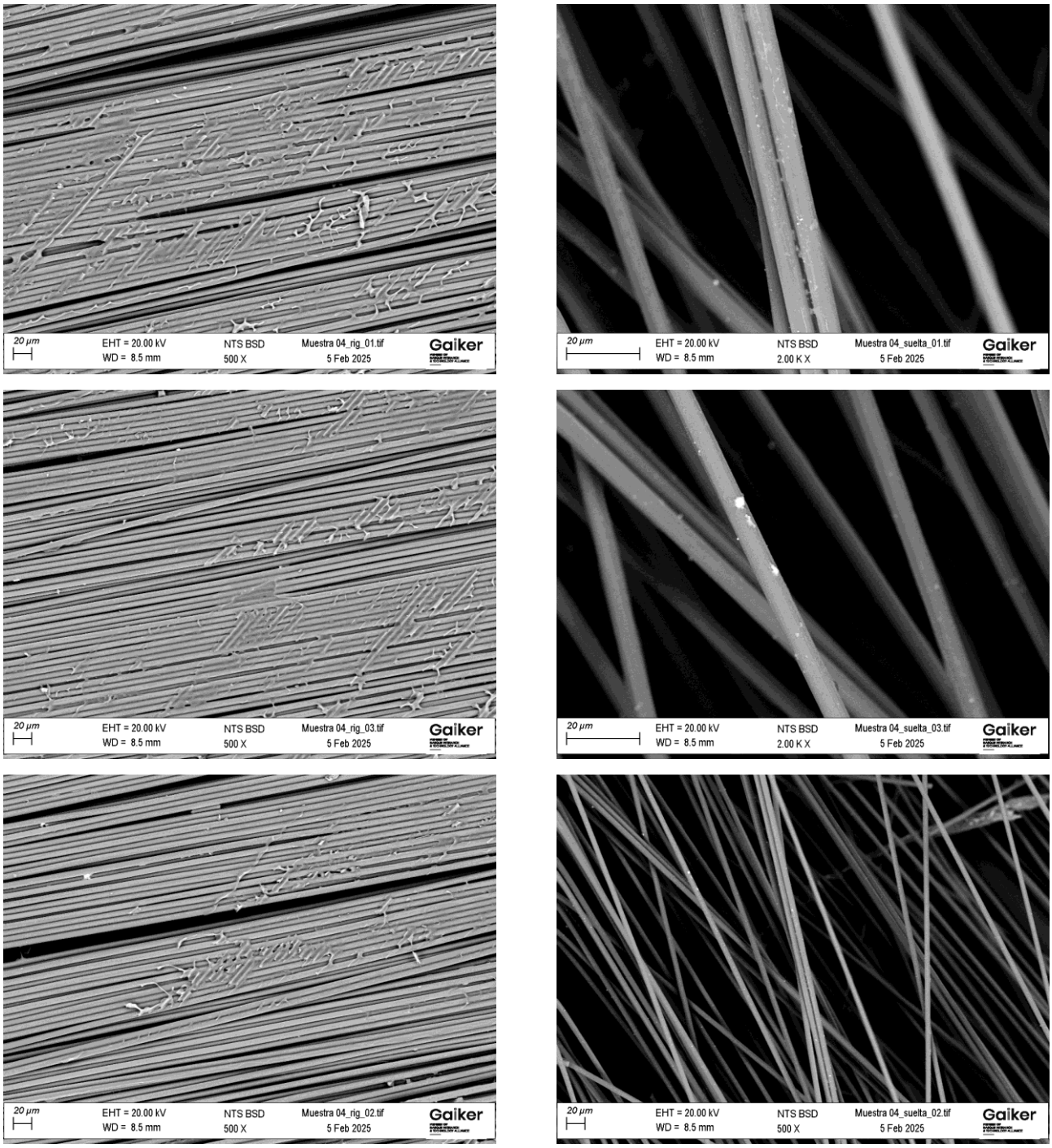


Figure 19. r-CF (P-O) SEM

The difference in the quality of the r-CF is due to the fact that airflow penetrates more effectively into the fibres located at the top of the reactor, resulting in more complete char removal. In contrast, the fibres positioned at the bottom of the reactor are exposed to higher temperatures, which causes overheating. As a result, these fibres develop a brittle structure with adhered residues.

In conclusion, it is proposed that the feeding of the sample into the reactor should be carried out properly in order to ensure the correct airflow circulation between the composites during the process to achieve homogeneous oxidation and preventing overheating.

r-MW characterization

Together with the r-CFs, the microwires inserted in the NCF used in the CFRP are recovered. To check its state three analysis were carried out.

First, a sample of the r-MWs was check on the SEM. As shown in Figure 20 the glass covering of the r-MWs has been affected by the oxidative pyrolysis process (or by manufacturing processes of the CFRP), although it seems that the core of the MW keeps in good condition.

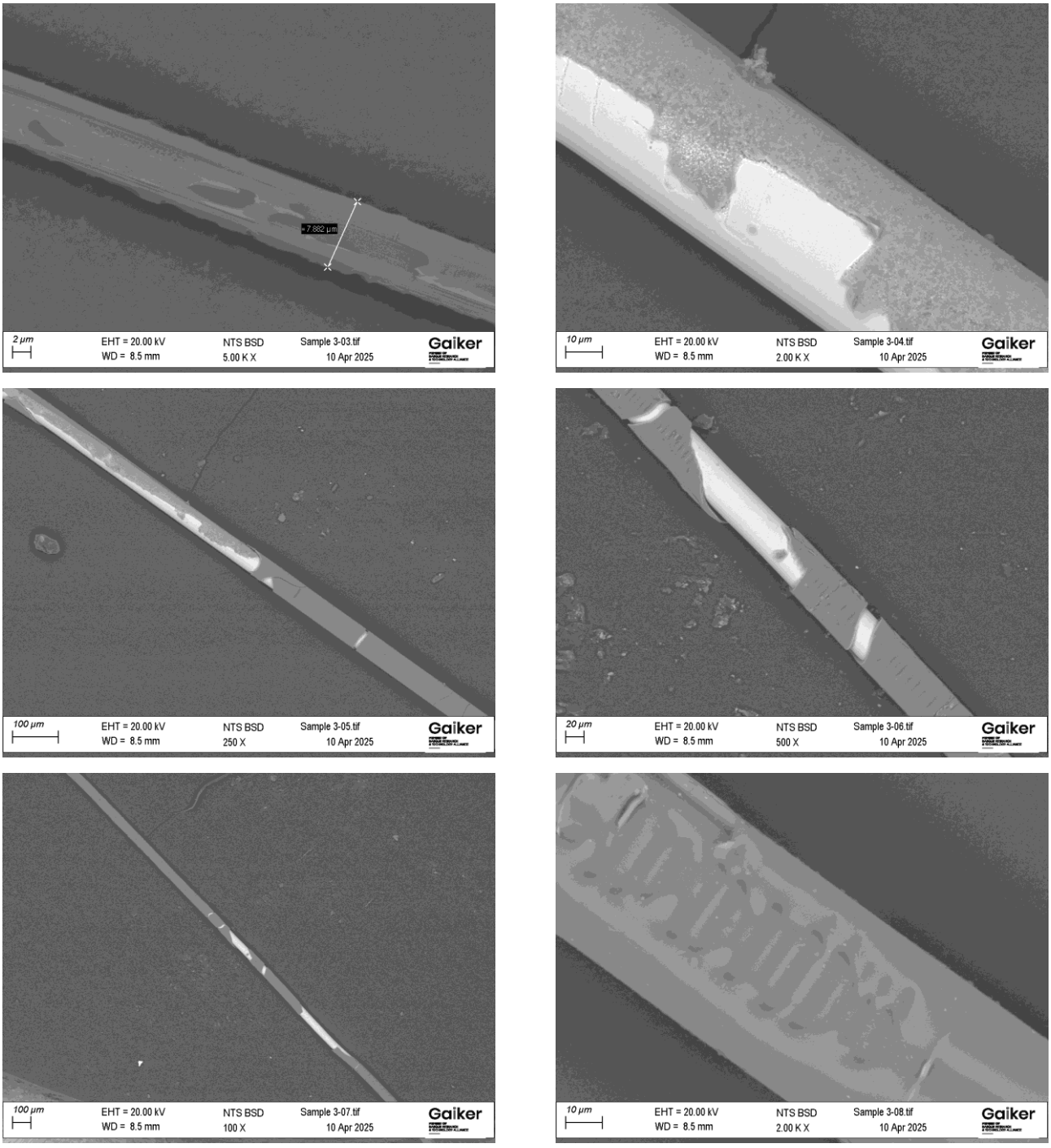
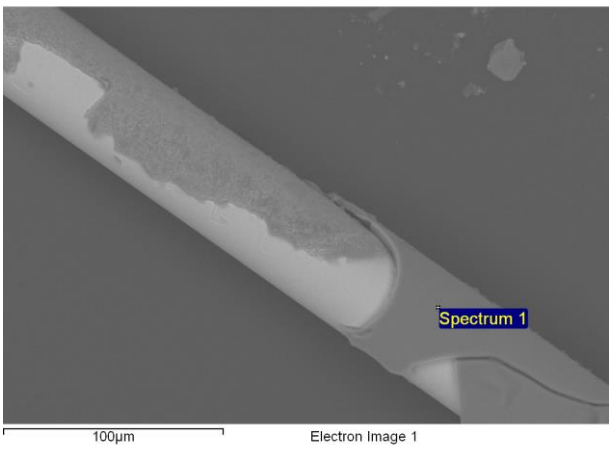


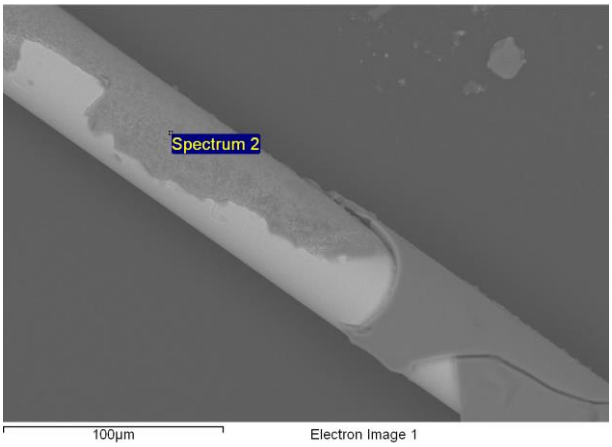
Figure 20. SEM images of the r-MWs

Secondly, taking advantage of the capability of the SEM equipment to carry out EDX analysis (Energy Dispersive X-ray spectroscopy) to analyse the elemental composition of the r-MWs, some tests have been carried out in different sections of the microwires. The results shown in Table 5 demonstrate that the formulation of the core part has not been affected.



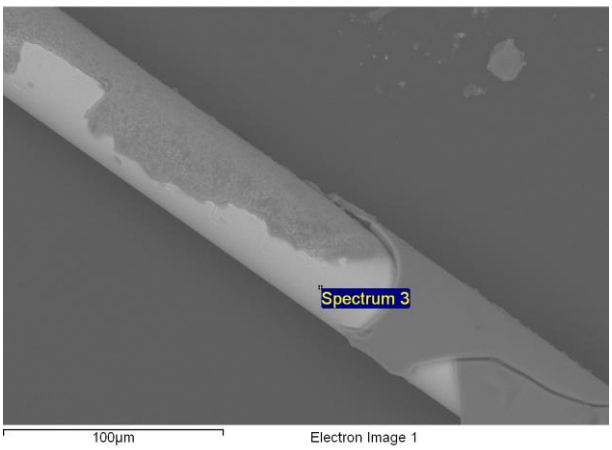
Spectrum 1: Glass covering section

Element	Weight%	Atomic%
O	38.94	53.20
Na	1.91	1.81
Al	1.69	1.37
Si	54.07	42.08
K	1.43	0.80
Fe	0.67	0.26
Co	1.28	0.47
Totals	100.00	



Spectrum 2: Mixed Glass covering – Core section

Element	Weight%	Atomic%
O	18.05	38.16
Na	4.88	7.18
Si	15.60	18.78
K	0.64	0.55
Mn	3.00	1.84
Fe	8.93	5.41
Co	44.04	25.27
Ni	4.86	2.80
Totals	100.00	



Spectrum 3: Core section

Element	Weight%	Atomic%
Si	10.75	20.11
Mn	0.71	0.68
Fe	5.20	4.89
Co	73.00	65.07
Ni	10.33	9.24
Totals	100.00	

Table 5.Results of the EDX tests

Thirdly, some tests have been carried out to check the GMI of the r-MWs. As shown in Figure 21 the values have increased significantly. This could be related with the crystalline structure of the alloy due to the high temperatures

of the recycling process and the residence time, which could have caused the original amorphous structure to a crystalline one, what is directly related with the GMI values.

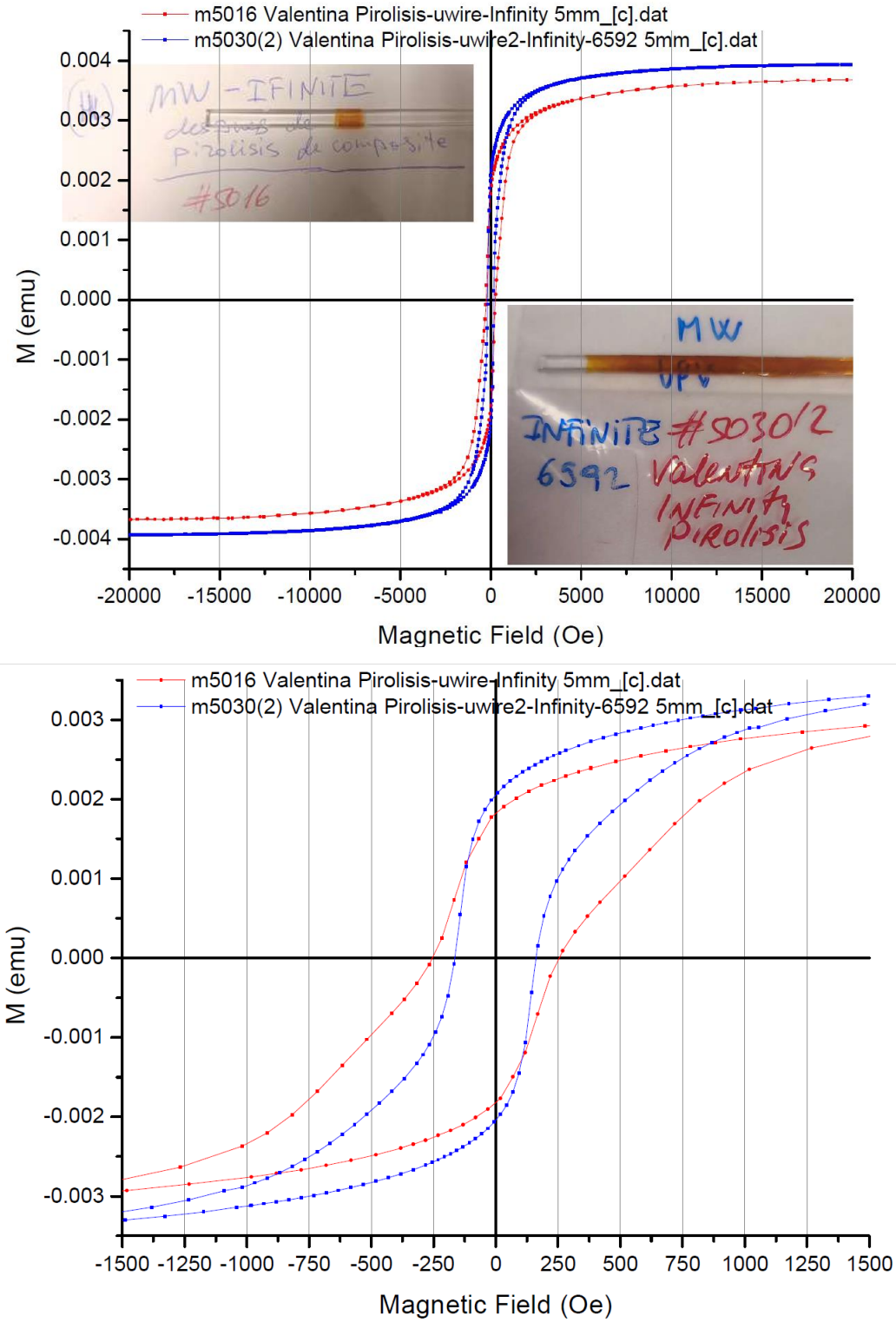


Figure 21. r-MWs GMI

r-CF characterization

Teijin carried out two types of single filament testing as a method to characterize the impact of the different pyrolysis treatments on the mechanical properties of the carbon fibre. The test methods included single filament tensile testing with the FAVIMAT AIROBOT II (company: Textechno) as well as single fibre pull-out testing performed with the FIMATEST+ (company: Textechno). The recycled carbon fibre samples were compared to virgin Tenax™-E STS40 E23 24K 1600tex fibre and Tenax™ STS40 F13 24K 1600tex S.

Filament tensile testing

To determine the tensile strength and modulus after undergoing pyrolysis treatment, Teijin performed filament tensile tests with an automatic single fibre tester (FAVIMAT AIROBOT II from Textechno). In total five samples were to be tested, two virgin carbon fibre samples and three recycled carbon fibre samples. The sample preparation of the recycled carbon fibre proved itself challenging as the samples were of relatively short length and bonded together after the process. Separating single filaments was difficult and for one of the three pyrolyzed samples it was not possible to attain single filament samples as the filaments were too brittle.

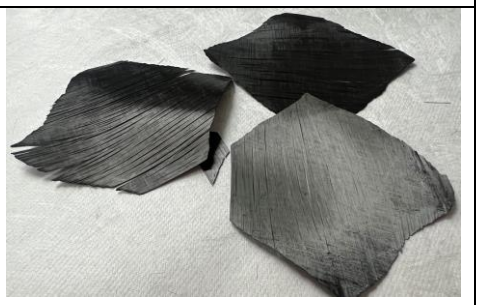
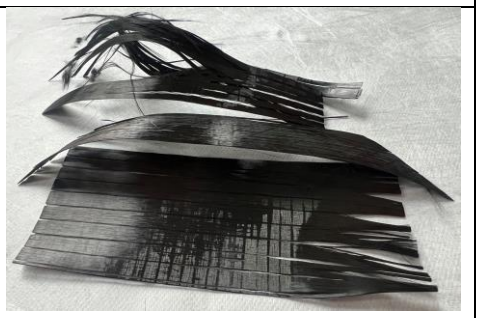
Sample name	Sample description	Exemplary image
rCF sample 1	Left-over laminate from IDEKO, not based on Teijin's NCF; fibre and resin unknown. It was used to determine the necessary parameters for the pyrolysis treatment.	
rCF sample 2 ("soft")	Laminate manufactured by IDEKO as part of the Infinite project; NCF based on STS40 fibre. Section of the laminate came out more loose and softer than others depending on the placement in the pyrolysis chamber.	
rCF sample 3 ("stiff")	Laminate manufactured by IDEKO as part of the Infinite project; NCF based on STS40 fibre. Section of the laminate came out stiffer and more brittle than others depending on the placement in the pyrolysis chamber.	

Figure 22. rCF sample overview

For the test the single filament samples were prepared and placed into the machine's repository. The samples then were tested automatically one after another. Each filament is placed into the testing unit, held by two clamps and then undergoes a standard tensile test.

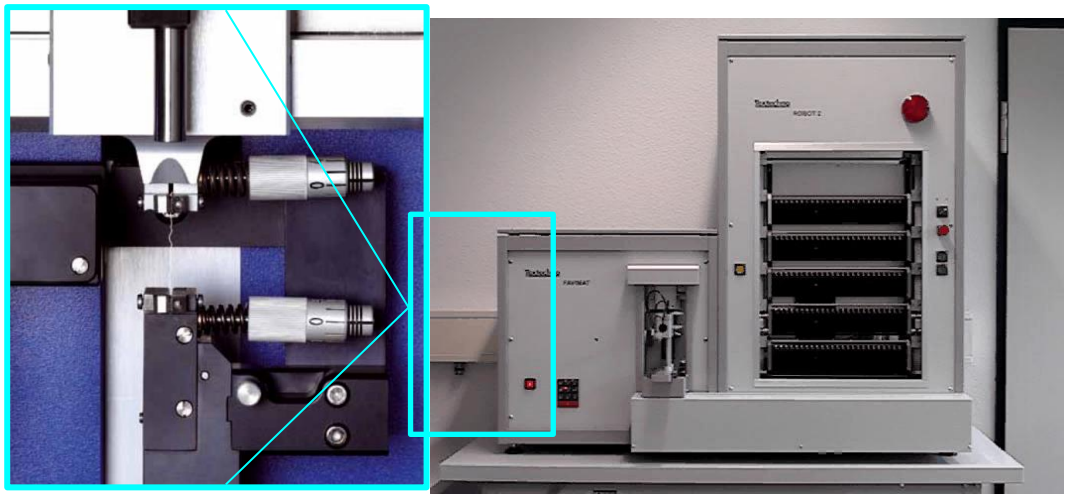


Figure 23. FAVIMAT AIROBOT II test machine

The results show lower values in tensile strength as well as elongation for all recycled samples compared to virgin carbon fibre. Between the three pyrolyzed samples, sample 3 was not able to be tested as it was too brittle. Sample one showed higher tensile strength and elongation compared to sample two. Sample two however, showed the highest values for tensile modulus compared to all samples including virgin carbon fibre. It should be stated that for sample 2 there were only 24 specimens obtained. Sample one and the virgin carbon fibre samples were tested with 60 specimens each.

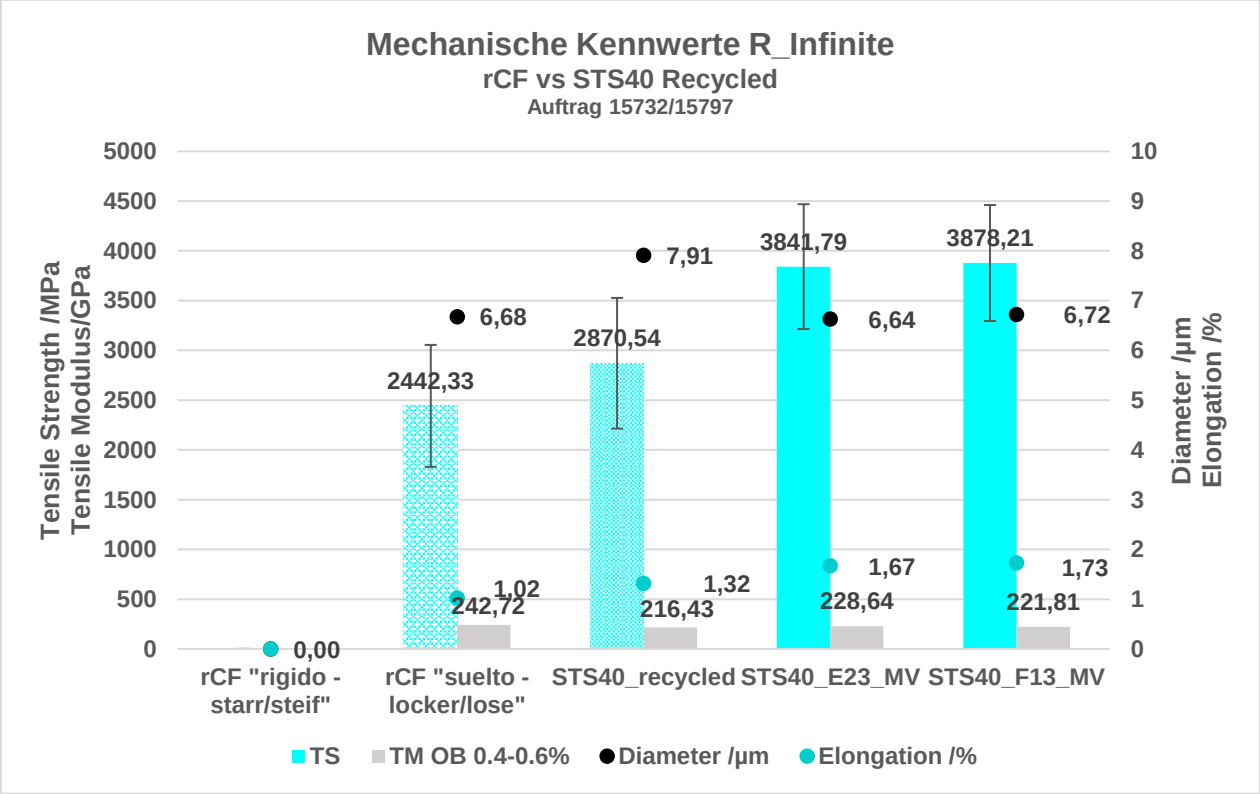


Figure 24. Results filament tensile testing

Single fibre pull-out testing

To gain insight into the interphase properties of a carbon fibre reinforced composite Teijin performed a single fibre pull-out (SFPO) test. For this test a single filament is embedded into a drop of resin. This sample undergoes a curing cycle followed by the pull-out test itself where the filament is being pulled out of the matrix. The surface finish of the carbon filament will influence the interlaminar shear forces in a composite as there is a direct link between surface finish and interfacial bonding with the matrix. Virgin carbon fibres have a polymer coating – called sizing – which helps to improve the bonding of the fibre with the matrix. After treatments such as pyrolysis, this sizing will be damaged or removed completely affecting the interfacial bonding. The test was performed with a FIMATEST+ from the company Textechno. The resin used in the performed tests was RTM 6. In total three recycled carbon fibre samples were to be tested; however, one sample did not allow for specimens to be obtained as it was too brittle. Virgin carbon fibres were not included as a reference here.

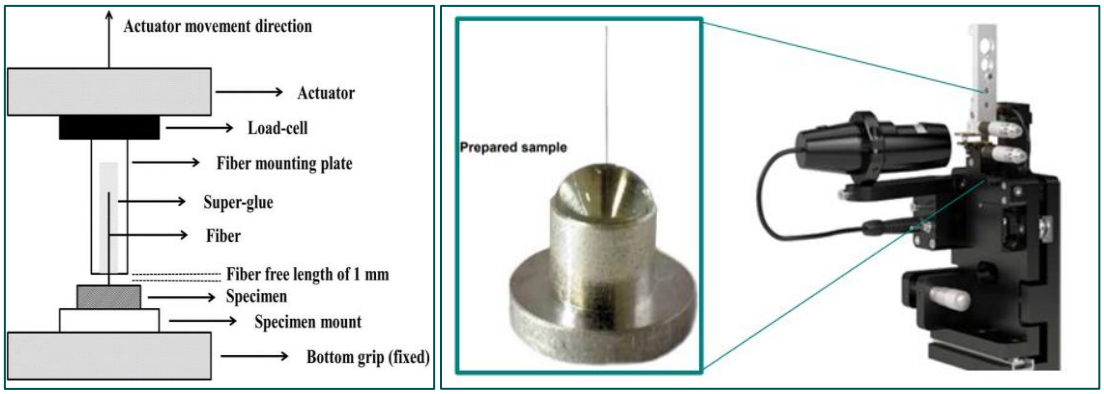


Figure 25. Sample preparation of single fibre pull-out test

For each of the tested samples 16 specimens were obtained. Sample one shows significantly higher values for all properties.

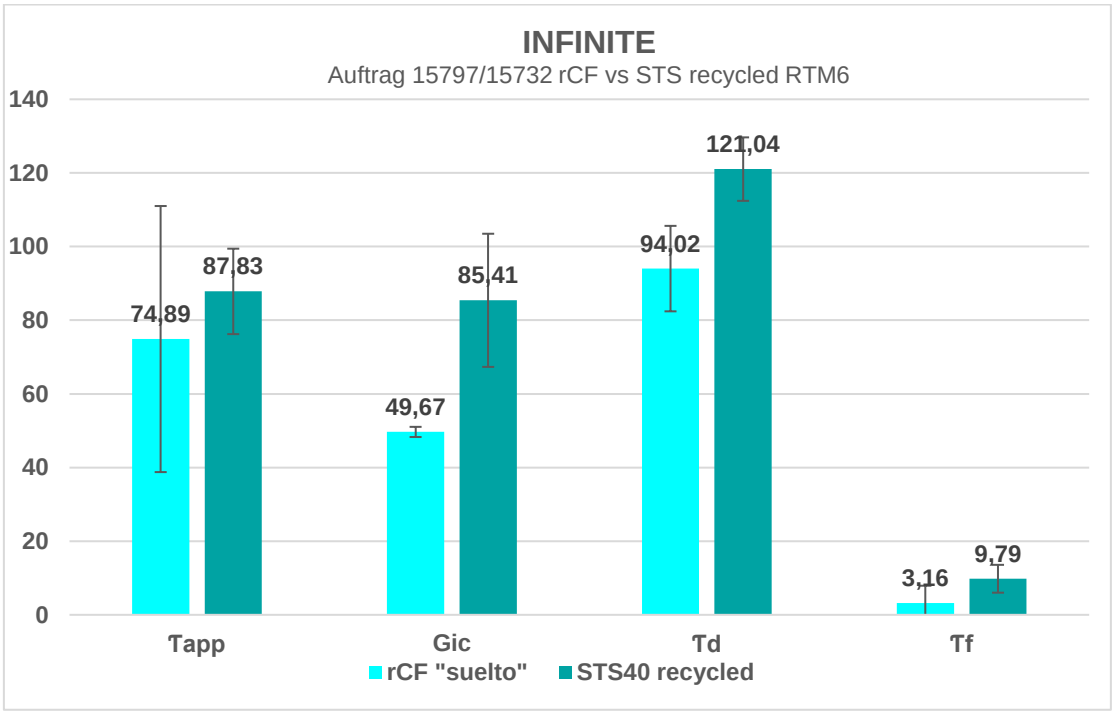


Figure 26. Results Single fibre pull-out testing

3.1.3 RESULTS

Filament Tensile testing

The measured diameters of the samples showed that the carbon fibre used in rCF sample 1 was made out of a different fibre type – not from Teijin as Teijin supplies fibres based on 7 µm and 5 µm filament diameters only. The diameter measurements for all other samples were on the same level. This supports the notion that all samples are based on the same carbon fibre (STS40). It also gives an indication that the pyrolysis treatment had no effect on the fibre that would result in a measurable degradation.

The tensile strength data of the performed tensile tests showed lower results for all recycled samples compared to the virgin carbon fibre samples. The recycled samples showed to be much more brittle in feel compared to the virgin carbon fibre making lower results in tensile strength plausible while tensile modulus results remained on a similar or even higher level.

While unclear what process parameters during pyrolysis result in this increased stiffness and reduced strength of the carbon fibre, the order of placement of the laminate samples in the pyrolysis chamber is most likely the cause for the uneven treatment of the fibres which resulted in the two different types of samples (rCF sample 2 and rCF sample 3) coming from the same batch undergoing the pyrolysis process. As the laminate pieces were placed very tightly in the chamber the medium flow in the chamber was uneven resulting in some laminate areas undergoing higher levels of treatment.

Vincent et al. (Jamin D.S. Vincent1, 2024) compared differently treated recycled carbon fibre samples with their virgin reference samples. Their work was based on carbon fibre provided by Teijin Carbon Europe and also included filament tensile testing. Their work included the comparison of HTS45, ITS55 and IMS65 and showed no significant drop in tensile strength for HTS45 however a significant drop for ITS55 and IMS65. Additionally, tensile modulus for HTS45 dropped while ITS55 and IMS65 tensile modulus remained on a virgin fibre level. Therefore, the effects of pyrolysis will be fibre dependent, but in most cases will result in reduced tensile strength.

Single fiber pull-out

The samples tested are of two different laminates and show no conclusive results. In order to evaluate the efficacy of this test method for measurement of recycled carbon fibre additional tests should be carried out including obtaining reference data of virgin carbon fibre.

Additionally, it is advised to test the micro wire with this test method as results obtained within the Infinite project from other project partners indicate that the integrated micro wires show a different level of fibre-matrix-adhesion compared to the carbon fibre which can then result in deviations during stress-strain-measurements based on the wires' signals.

3.2 RECYCLING OF CARBON FIBRES (TCE)

The use of Liquid Composites Moulding (LCM) technology in the Aerospace industry is taking more a more presence against legacy prepreg technology. One of the draw backs from using LCM technology, with Non-Crimp Fabrics (NCF), is the waste generated during production, making the buy-to-fly ratio less attractive. To overcome this problem, the industry needs to address recycling solutions for this stage of the life of the part as well. This section emphasises the recycling opportunities of the material before it reaches the end-of-life. Those opportunities arise during the manufacturing of the part, including raw material production.

The recycling of carbon fibre has become increasingly important due to environmental concerns, regulations, and the economic benefits of reusing this valuable material. The proposed solution presented on this section aims to address the challenges associated with effectively using recycled dry carbon fibre textiles by developing a converting technology to generate a high-value product that can be incorporated back into the supply chain.

The recycling processing of dry carbon fibre textile waste is shown in Figure 27. This waste/scrap is originated at different stages of the material flow:

a) Carbon Fibre Raw Material Supplier: This carbon fibre waste generated during CF production is material that has gone through the full production process but has not reached the end of the process. The waste material is Carbon fibre filaments with the sizing, but randomly entangled.

b) Downstream Material supplier- NCF production: two types of waste are generated during the NCF production: trimmed edges of the NCF, which are short carbon fibre filaments with not stitching or binder on it; and NCF that did not meet the quality criteria. On this case the scrap NCF contains additional elements such as the stitching yarn, powder binder, and the microwires (if sensorised-NCF).

c) Part Manufacturer: part production processes using NCF material involve different steps that can generate waste/scrap material before the infusion is done:

   a. Ply Cutting: the NCF is provided in wide rolls from where the plies are cut to the final ply geometry. This process generates off-cuts that cannot be use on the final part and are consider waste(off-cuts). This off-cuts will contain the additional elements such as stitching yarn, powder binder and if sensorised, microwires.

   b. Consolidated preform trimming: once the full stack of plies is put together and the binder is activated, preform, sometimes it will be required additional trimming to achieve the final shape of the part. This is especially relevant for net-shape close mould processing. This waste is the same as from ply cutting, but the binder is activated.

d) End-of-life: on this case, when the composite part reaches the end-of-life, it goes through a more complex recycling process, pyrolysis, where the matrix is removed, generating a final product that is dry carbon fibre. This process has been explained in Para 4.2 Chemical recycling: pyrolysis. The final product of this process is the dry carbon fibre without stitching yarn, powder binder or sizing.

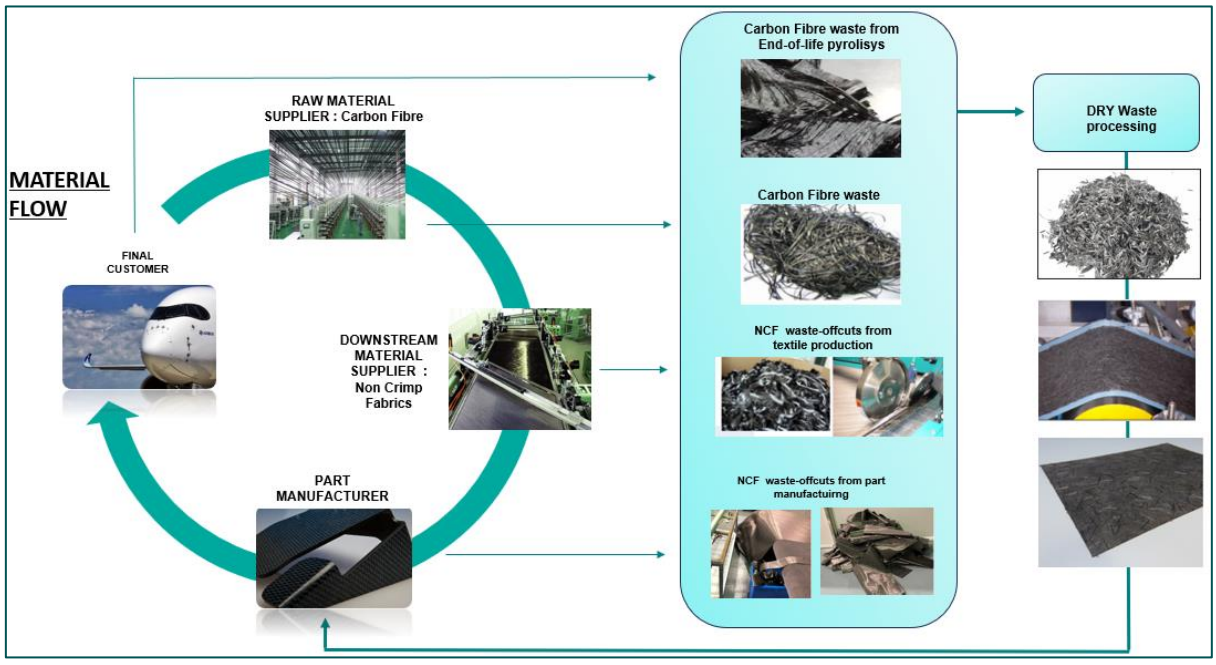


Figure 27. Example of material flow of a carbon fibre composite part for Aerospace application, and waste generated at different production stages.

All the above different stages of the material flow have in common the generated waste: dry carbon fibre. However, depending on feedstock, the dry carbon fibre will require a different approach to achieve a recycled product.

This section will be focused on the recycling process of NCF (from downstream material supplier and part manufacturer).

DRY NCF WASTE PROCESSING

The aim of this section is to show a solution for the NCF waste that is generated before the composite layup is infused with the resin system:

- Production scrap: this is generated during raw material production
  a) Non-conformity NCF
- Post-production scrap: generated during product processing
  b) During ply cutting/deposition
  c) During trimming

The proposed solution aims to demonstrate a fully integrated process flow based on recycling the NCF waste from production by transferring into a CF non-woven. CF non-wovens are randomly oriented short carbon fibres in a mat (ranging from 50 g/m^2 to hundreds) that can be used as a lightweight component in many industries, such automotive, aerospace, industrial and construction. Using rCF (recycled CF) for the construction of the non-woven veils is a sustainable solution that supports circular economy.

In the case of this project, a rCF non-veil have been produced and incorporated into an NCF, proving the close loop of reintegrating the recycled material into an NCF.

For economical-time feasibility within this project the focus has been on NCF without microwires. The reason for this was the conversion process requirement of minimum quantities of 500 kg of scrap NCF to run the equipment, which was not available. However, the presence of MW should not influence the converting process.

The NCF off-cuts recycling flow is as shown in Figure 28:

Figure 28. Example of process flow: form collection of the NCF offcuts to conversion into a non-woven.

1. NCF material off-cuts: all the material scraps are collected from customer facilities. Transport can be done on large containers or bags. There is no need to sort the material.
2. NCF cleaning (heat treatment): this step can be optional. The intention of cleaning the material is to remove the stitching yarn, binder, sizing, so the residue is only CF and potentially microwires. If the material is not submitted to a cleaning process, the results would be a non-woven rCF with all the additional elements, which would impact the mechanical properties, acting as contamination. However, depending on final application of the rCF non-woven, mechanical properties could be sacrificed on behalf of better economical solution.

3. Material chopping: this stage is very challenging, as the material needs to be chopped to a regular length (6-10mm). The chopping length will be dictated by the non-woven process.
    a) If the NCF contains MW, there could be an additional step needed to separate the MW from the CF. However, MW could also be left on the non-woven as contamination.
4. Non-woven (rCF) processing: this step uses needle-punch dry non-woven processing, which starts with the short rCF laid out in a web formation, typically done by a machine such as a carding machine, which combs the fibres into the desired pattern. After this, barbed needles are punched vertically through the web to hook and entangle tufts(clumps) of fibres. This process helps to interlock the fibres and create a stronger fabric. The needles penetrate beyond the surface to form loops on the back, which can be left uncut or cut and brushed to give the desired surface appearance.

3.2.1 TESTS

The main objective of the test done by Teijin was to produce an NCF with a rCF non-woven veil. This is an important step to demonstrate how future material scrap can be converted into a high-value product and reintegrated into an NCF.

This trial focused on demonstrating the process, but had some limitations:

- Since the minimum material quantity to process the non-woven was 500 kg, virgin CF was used instead of rCF, as the high quantities were not available.
- However, according to Teijin's experience, using rCF on the same process has been proven.
- It has not been tested how the presence of the MW on the rCF offcuts can influence the recycling process.

This trial started with chopping the CF (external company: Altex), and then producing the non-woven via wet-lay process (external company: Tenowo):

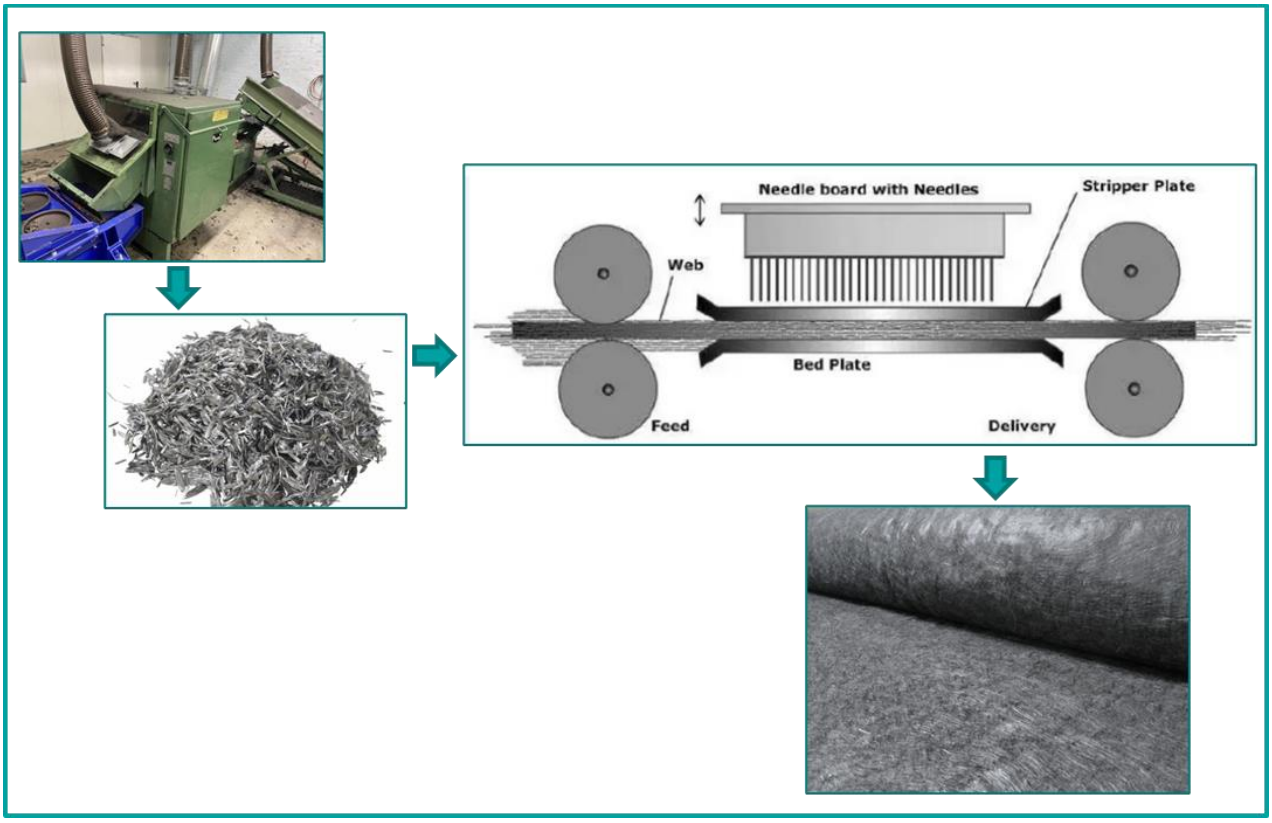


Figure 29. Process flow of non-veil production

The resulted no-woven roll was then sent back to TCE, where it was integrated into a NCF. The non-woven is stitched on the top layer of the NCF (Figure 30):

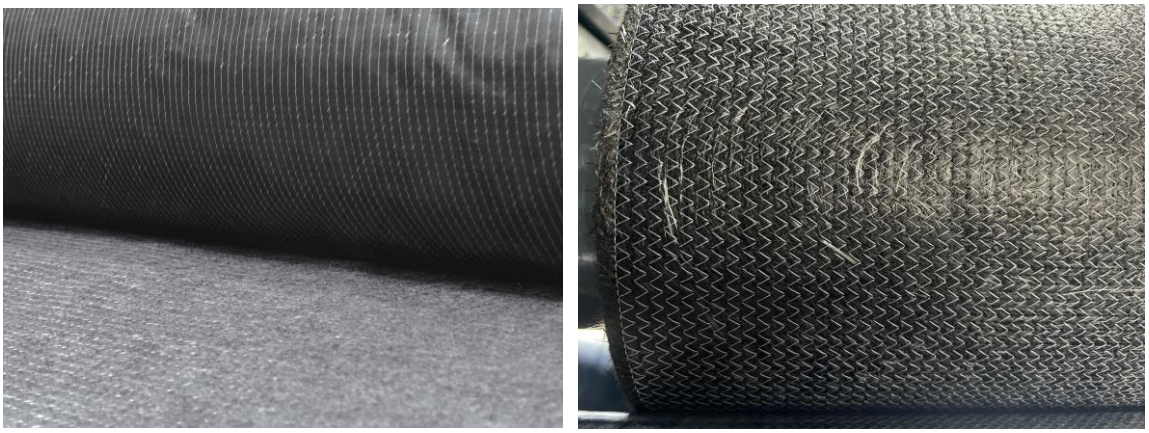


Figure 30. 300 g/m^2 NCF with embedded 200 g/m^2 rCF non-woven

[710223 X01_Tenax™ DRNF HT BD 0500 710223 X01 TDS reference]

3.2.2 RESULTS

The resulted roll of the NCF with the embedded non-woven passed the quality inspection at Teijin and was sent to Ideko. The intention was for Ideko to create a Laminate with this material and MW integrated manually to check the effect of the signal. However, due to time limitations this test was not performed.

3.3 IDENTIFICATION AND SEPARATION OF MICROWIRES IN A RECYCLED CARBON FIBER COMPOSITE (GAIKER)

The objective of the study was to evaluate the ability of intelligent identification techniques to identify the microwires (MWs) present in a carbon fibre composite (CFRP). For this purpose, two advanced analysis technologies were used: hyperspectral imaging (HSI) and laser-induced breakdown spectroscopy (LIBS). These techniques made possible to assess the feasibility of detecting the MWs for possible future reuse processes.

3.3.1 TESTS

Analysis by HSI

The system used by GAIKER consists of a HSI camera, a sample translation platform, an autonomous illumination system, a structure with a guide for height adjustment of the camera, and a PC with software for hardware control and image acquisition and analysis (Figure 31).

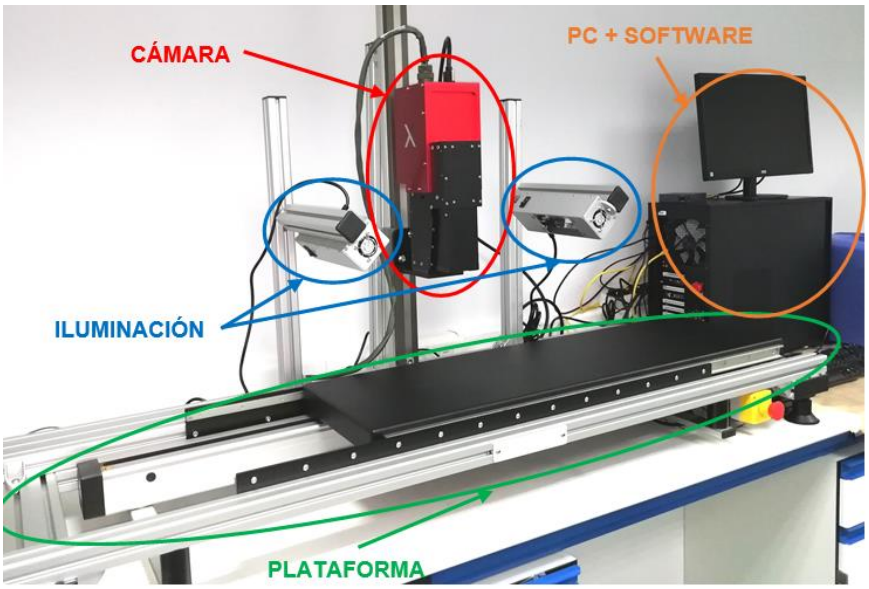


Figure 31. Image of the HSI system used at GAIKER.

The HSI camera used is of the push-broom or linear type, which obtains images by scanning, and covers the wavelength range of the short-wave infrared spectrum (SWIR).

The illumination system consists of two 150 mm 100 W halogen lamps. To achieve optimum resolution and image intensity, the position of both the camera and the two luminaires and the integration time have been adjusted. The final focusing height of the camera was 270 mm, obtaining a FOV of 95 mm. The luminaires were positioned symmetrically with respect to the camera axis at 480 mm distance from each other, at 240 mm height and an approximate 45º tilt.

The configuration of the system used required careful adjustment, since the composite, being black, had a high level of absorption in the SWIR spectrum. The initial hypothesis was that this characteristic could make it difficult to identify the microwires sewn into the material. However, the objective was to determine whether, despite the overall absorption of the composite, it was possible to distinguish these microwires in the hyperspectral image.

After starting up the system and scanning the sample, it was observed that, although the composite appeared as a homogeneous and dark surface with hardly any signal generation in the hyperspectral image, the stitched microwires could be slightly differentiated. Figure 32 illustrates this phenomenon, where it can be seen that the detection of the microwires was possible as long as the camera was focused on the side of the sample where the microwires were superficially stitched. On the contrary, when the sample was scanned from the opposite side, the microwires were not visible, as shown in Figure 33.

Figure 32. Sample of the complete composite scanned from the microwires side.

Figure 33. Sample of the complete composite scanned from the side opposite to the microwires.

In addition to the analysis on the original composite, a second experimental phase was performed after subjecting the samples to a pyrolysis process. During this treatment, the resin and polyamide originally present in the composite were removed in the pyrolysis process, which made it possible to expose the microwires in more accessible layers. The resulting samples were then scanned to assess whether the microwires could be more clearly identified.

As presented in Figure 34Figure 34, hyperspectral analysis of a layer obtained after pyrolysis also revealed the presence of the microwire. An additional test was performed with singular microwire to check the ability of the system to detect it without interference from the composite material. Figure 35 shows the microwire marked with a signal for identification, and in Figure 36 when zoomed in, the microwire can be seen to be distinguishable.

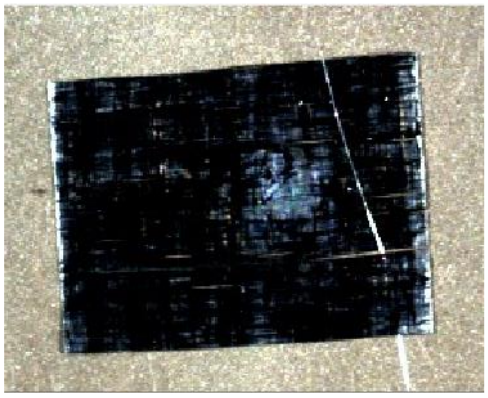


Figure 34. Composite layer with microwire after the pyrolysis process.

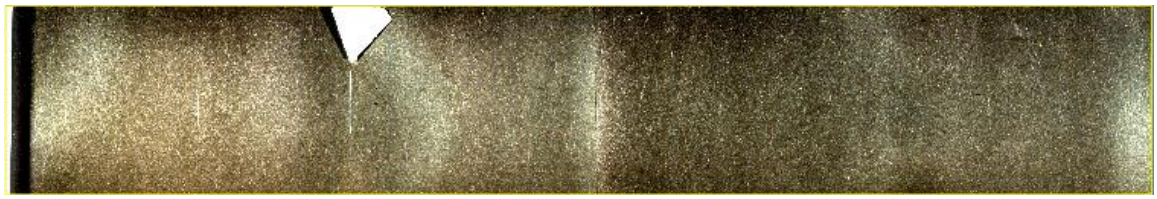


Figure 35. Scanned microwire.

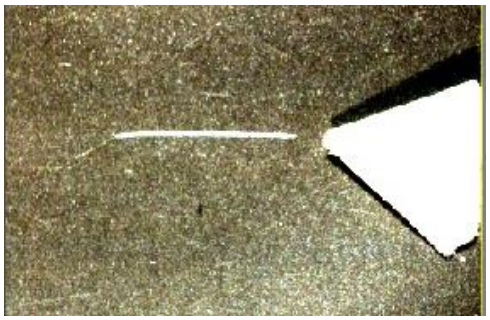


Figure 36. Scanned microwire with zoom.

The results obtained indicate that, although the resolution of the HSI system is limited, it is possible to detect the microwires both in their sewn state within the composite, as long as they are in a superficial position, and in the samples after the process of pyrolysis. For future research, it would be interesting to develop identification and classification models trained with different pre-treatments and analysis algorithms, in order to improve the segmentation and concentration of samples containing microwires. In addition, since the HSI technique allows this initial detection, it would be necessary to implement a second system for the separation and recovery of the microwires. Taking into account the microwire's composition and the magnetism they present, a possible solution could include a magnetization mechanism to facilitate their extraction once identified.

Analysis by LIBS

To complement the study, an elemental analysis technique, such us laser-induced breakdown spectroscopy (LIBS), was used. This methodology was used to determine whether the microwires, composed of an alloy of cobalt, iron, boron and silicon with the atomic formula $Co_{72}Fe_4B_{13}Si_{11}$, could be detected by laser ablation.

The LIBS system used by GAIKER was integrated into a conveyor belt, which enables online scanning and analysis of samples (Figure 37Figure 37). The LIBS analyser consists of two spectrometers covering different wavelength ranges (spectrometer 1: 230.295 - 501.264 nm and spectrometer 2: 744.986 - 941.484 nm), a laser head installed on the conveyor belt and a laser source emitting a radiation of wavelength 1,064 nm. The experimental system is completed by a specific software with applications to adjust the measurement parameters, obtain LIBS emission spectra, apply pre-treatments to these spectra, train and evaluate calibration models based on multivariate data analysis algorithms ("machine learning" models) and operate on-line to classify materials from the analysis of their respective spectral fingerprints (LIBS signal analysis).

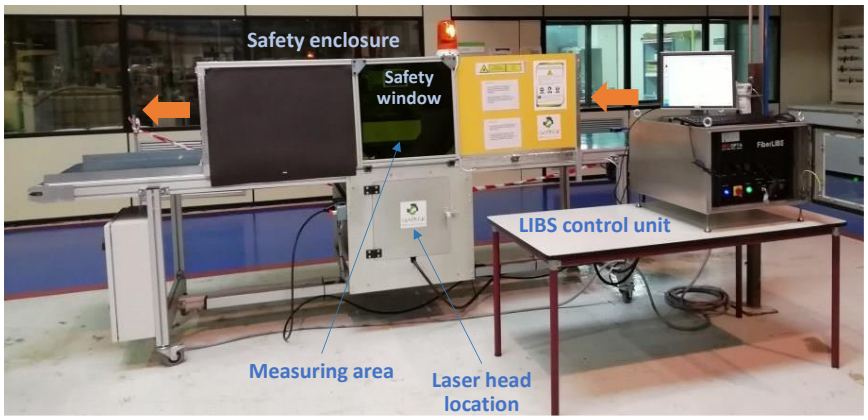


Figure 37. Pilot automatic identification system based on LIBS technology. (GAIKER)

Two types of samples had been used: layers with microwires and layers without microwires, all of them previously subjected to a pyrolysis process. The intention was to evaluate whether the characteristic elements of the microwires generated differentiated signals in the spectra obtained. However, the results showed that the signals coming from the composite layers or fibres were extremely weak. During the spectroscopic analysis, we proceeded to evaluate the spectra individually, comparing the spectral fingerprints of the different samples and focusing on the specific wavelengths at which the microwire elements should give signal (Co, Fe, B and Si).

Table 6 presents the data obtained, showing for each element and its respective wavelength the comparison between the spectra of the samples with microwires (represented in pink) and the samples without microwires (represented in green). In addition, in the last row of the table, figures representing the average of the spectra obtained for each type of sample are included: on the left, the complete spectrum of the samples is shown (the two spectrometers) and on the right, the first part of the spectrum is shown (first spectrometer) where all the metals present in the microwire appear. As it can be seen, no significant differences were found neither in the full spectra nor in those focused on specific wavelengths. This indicates that LIBS, at least with the characteristics used in this study, does not prove to be a suitable technique for the identification and classification of microwire samples. Unlike HSI, this technique does not seem viable as a prior method of concentrating samples with microwires for subsequent separation.

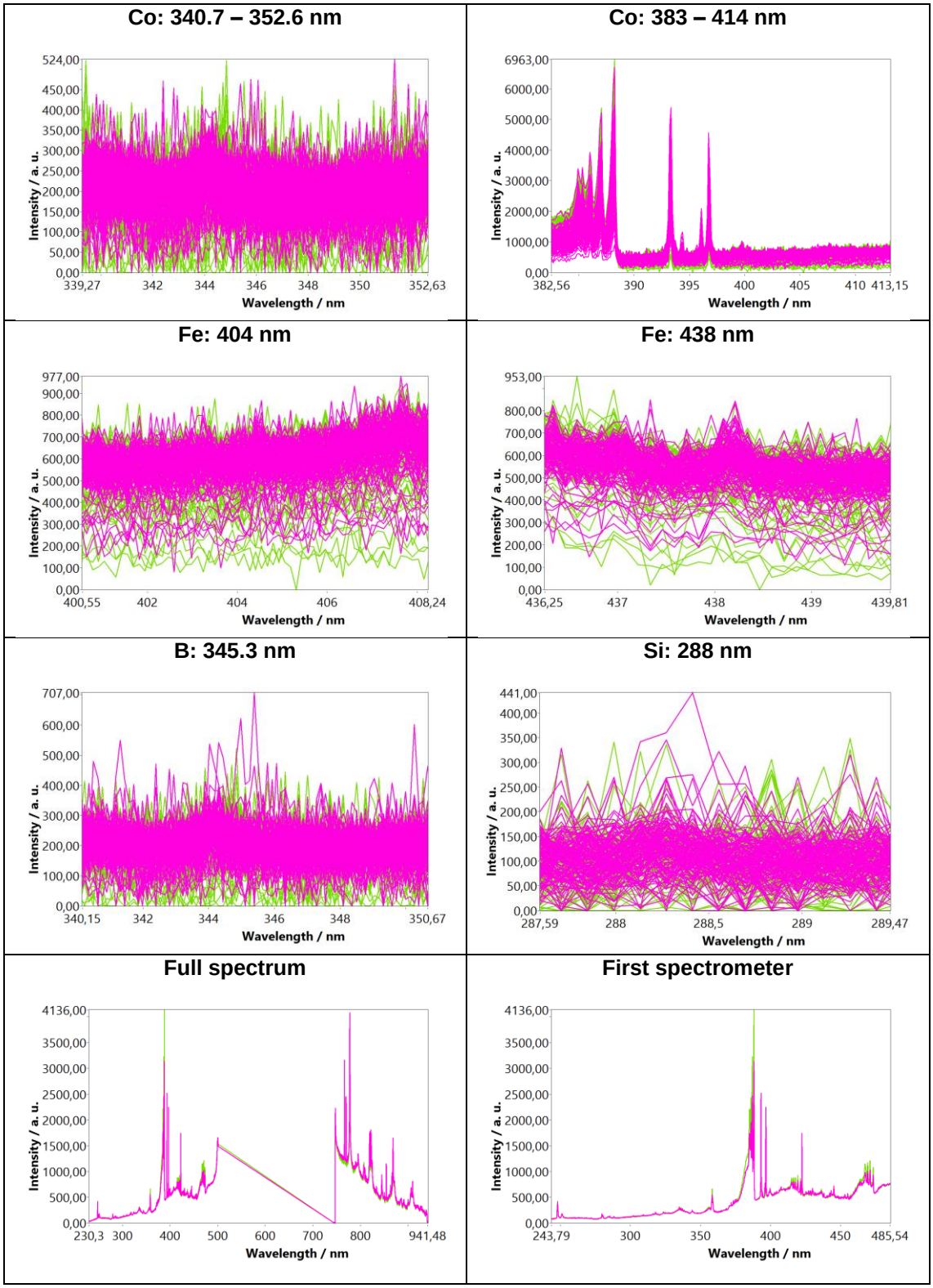


Table 6. Images of the spectra obtained at each wavelength of each element. Additionally, in the last row, the whole spectrum of the analysed samples and the first part of the spectrometer, where all the metals present in the microwire are shown

3.3.2 RESULTS

The results obtained indicate that, for the moment, the best strategy for the recovery of the microwires is through the use of a magnet, since the analysed microwires ($Co_{72}Fe_4B_{13}Si_{11}$) present magnetic properties that facilitate their collection.

In this context, the HSI technique does seem to have potential as a preliminary method for the identification, classification and concentration of samples containing microwires. From this classification, such samples could be subjected to a secondary recovery process based on magnetization. Tests carried out with a magnet have shown that it is perfectly capable of attracting and recovering the microwires present in the analysed samples.

Therefore, future research could focus on optimizing the use of HSI to improve its identification and segmentation capabilities, thus allowing the implementation of an efficient recovery process based on magnetic separation.

4 REFERENCIAS

A. Zhukov, P. C.-L.-L. (2022). Advanced functional magnetic microwires for technological applications. *J. Phys. D: Appl. Phys, 55*, 253003. doi:10.1088/1361-6463/ac4fd7

Churyukanova, M., Zhukova, V., Talaat, A., Kaloshkin, S., Kostitcyna, E., Shuvaeva, E., . . . Zhukov, A. (2014). Correlation between thermal and magnetic properties of glass coated microwires. *J. Alloys and Compounds, 615*, S242.

D. Makhnovskiy, A. Z. (2008). Tunable and self-sensing microwave composite materials incorporating ferromagnetic microwires. *Advances in Science and Technology, 54* , 201-210.

Esfandiar Pakdel, S. K., Pakdel, E., Kashi, S., Varley, R., & Xung. (2021). Recent progress in recycling carbon fibre reinforced composites and dry carbon fibre wastes. *Resources, Conservation and Recycling, 166*.

Jamin D.S. Vincent1, A. X. (2024). ENSURING MEASUREMENT CONFIDENCE IN RECYCLED CARBON. *Proceedings of the 21st European Conference on Composite Materials*, Volume 7 - Life cycle performance [78-83].

L. Panina, M. I. (2011). Tuneable composites containing magnetic microwires. En *Metal, ceramic and polymeric composites for various uses* (págs. 431-460). doi:10.5772/21423

Rani, M., Choudhary, P., & Krishnan, V. (2021). A review on recycling and reuse methods for carbon fiber/glass fiber composites waste from wind turbine blades. *Composites Part B: Engineering, 215*.

V. Zhukova, A. C. (2001). Tailoring of magnetic properties of glass coated microwires by current annealing. *J. Non-crystalline solids, 2001*, 31-36.

V. Zhukova, M. I.-L.-G.-E. (2024). Free Space Microwave Sensing of Carbon Fiber Composites With Ferromagnetic Microwire Inclusions.

Zhukova, V., Cobeño, A., Zhukov, A., & de Arell, A. (s.f.). Correlation between magnetic and mechanical properties of devitrified glass-coated Fe71.8Cu1Nb3.1Si15B9.1 microwires. *J. Magn. Magn. M*.

5 CONCLUSIONS

Recycling of CFRP production and end-of-life wastes is becoming increasingly important due to the growing use of carbon fibres as reinforcement in high performance composites. Recycling of production waste has, currently, the highest priority, like up-cycling of production by-products. This work package covers both solutions simultaneously, since the end products and processes of both feedstock materials are very similar, proving the following conclusions:

Reuse of sensorised CFRP:

1. One of the main constraints for reusing CFRP panels or parts is the difficulty of knowing their actual condition, “health” of the panel/part, but the sensorization of these CFRP panels/parts with the developed MWs, can make easier this evaluation.
2. Once a reliable correlation between MWs magnetic signals and the state of the CFRP products, understood as the indirect measurement of their mechanical and thermal properties, can be developed the reuse strategy, to enlarge the life span of these products, could be feasible in a cost-efficient way.
3. The final reuse application could be determined according to the properties correlated with the inspection of the CFRP products with the equipment developed in the project.

Chemical recycling process of the CFRP waste:

1. The pyrolysis test demonstrated that this chemical recycling alternative is technically feasible although some conditioning of the rCF would be needed before applying the process.
2. The pyrolysis-oxidation process demonstrated to be a feasible route for recycling both CFs and MWs from the pyrolyzed CFRP waste. In this case, nor or fewer conditioning of the rCFs would be needed.
3. The rMWs obtained in the combined pyrolysis-oxidation process, could be used to take advantage of their magnetic properties although in a different application due to the change in their GMI properties from the virgin MWs used in the production of the sensorised CFRP.

Recycled carbon fibre:

1. Efficient use of recycled carbon fibre: the technology will enable the efficient use of recycled carbon fibre, reducing waste and supporting sustainability.
2. Cost savings: By achieving a lower converting cost, we could provide a cost-effective solution for the industry.
3. Environmental benefits: this technology will contribute to the reduction of carbon fibre waste, supporting a more circular economy.
4. It is the clear target to maximize the use of side-products (or pre-consumer scrap) and post-consumer waste which will reduce the production of virgin materials and achieve CO_2 emissions reduction.