Fuctionalised Innovative Carbon Fibres Developed from Novel Precursors With Cost Efficiency and Tailored Properties

Final Report Summary - FIBRALSPEC (Fuctionalised Innovative Carbon Fibres Developed from Novel Precursors With Cost Efficiency and Tailored Properties)

Executive Summary:
Engineers are constantly pushing the boundaries, aiming to design composites structures by carbon fibers (CFs) with reduced cost, as well as, with improved mechanical properties. After more than 5 decades of research, a growing industry with a multitude of applications has been established and the need on new precursors that not derive from petroleum has been revealed. This requires a novel approach that introduces the significant challenge of production of CFs from “green” precursors and manufacturing of fibre-reinforced composites via ecofriendly-production techniques. FIBRALSPEC developed Innovative processes with streamlining and improved control, through Unit for Continuous PAN-based Carbon Fiber
Pilot Production. Green precursors of CFs based on lignin based blends have been synthesized since lignin has attracted significant attention as an alternative carbon source for conversion to CFs due to material’s abundance, high carbon content, low cost as byproduct of paper industry and renewable nature. The quality of polymer fibers spun by lignin depends on the spinning method and chemical modification of raw material. However, the improvement of lignin fiber spinning technologies has now reached a limit and new approaches are necessary to be developed based on chemical modification and functionalization. FIBRALSPEC also investigated the enhancement of mechanical performance of CFs composites by changing their structure, physical and chemical properties for enhancing of their service characteristics and opening of new functional possibilities of their applications. Different types of plasma processes were implemented for the surface treatment of carbon fibres, in order to analyse their advantages and disadvantages. Within this framework electrochemical treatment and subsequently electropolymerization onto CFs surface was applied in order to improve adhesion between CFs and matrix through wetting improvement. The modified fibers exhibited improved wetting properties in epoxy resin whereas the resulting modified carbon fiber composites demonstrated a significant increase in the ultimate tensile strength revealing enhanced bonding at the fiber matrix interface compared to the composite with pristine CFs. FIBRALSPEC also addressed the issue of production of CFs with enhanced properties by nanoprofiling and nanomodification of their surface using nanoparticles produced from plant raw materials. With nano-fibres becoming an increasing field of interest, ability to control and align the fibres produced during the electrospinning process has emerged as an area that has received much interest. A novel approach of electrospinning was applied and a unique mechano-electro-spinner (MES) has been designed, constructed and commissioned with a potential to be used in fields of application such as Carbon Fiber Industry, Polymer/Composite industry and textile industry. The work on the fabrication of Prototypes of advanced flexible supercapacitors was realised in different steps including determination of the specific concentration of carbon nanofibers (CNFs) mixtures (produced within FIBRALSPEC) to achieve the best results, fabrication and testing of the produced devices. The technology for Supercapacitors based on CNFs has been already up-scaled within the project and practices of interest for electronics, aviation and automotive industry have addressed during the last four years. The existing concept “Rapid Deployment Secure Emergency Shelter-FibreGlass” (RDSES-FG) which is designed and developed by applying traditional composite manufacturing techniques has been further exploited; employing the use of carbon fibre, the unit mass has been reduced while at the same time increasing rigidity, durability and end user usability, resulting in Rapid Deployment Secure Emergency Shelter Carbon Fibre (RDSES-CF). As for recycling and used of recycled CFs, new techniques have been used to provide commercially-relevant products that are manufactured from waste carbon fibres. Mathematical modeling was conducted so as to determine properties of CFs and composites, together with cost modeling; life cycle assessment assisted possible commercial risks that were continuously estimated during the project and quantify/assess the environmental impact of the used materials.

Project Context and Objectives:
Carbon Fibre composite materials are becoming nowadays more and more part of our lives. After more than five decades of research, CFs and their composites have reached maturity and they are currently not just a ‘high-end’ costly solution for low rate production, but represent a growing industry with a multitude of applications. The CF composites are key elements of smart material systems and structures.

Properties such as light weight, high strength, low stiffness, smartness, their dual use and more, makes these materials more and more attractive to various industrial sectors such as transport, electronics,
construction, and many other sectors.

Their success is due to their high strength-to-weight ratio and to the fact that in composites they exhibit a combination of valuable properties. In 2010 their production was around 75,000 tons per year, with an expected growth rate of around 7% for the following five years. Some of their most important applications are in the sports and leisure industry with articles for many sports (tennis racquets, golf clubs, bicycles etc.), in the aerospace industry (the newest and largest commercial airplanes, Boeing 787 and Airbus A380, have a large part of their airframe built using carbon fibre composites) and for the blades used in wind turbines. The most important precursor (in terms of production volume) is polyacrylonitrile (PAN) fibres; PAN-based carbon fibres represent more than 70% of the total CF production. A point to note here is the supply and availability of CFs in the future where there is a need for the EU States to be independent of the current supply chain. Moreover, there is also a need to consider precursor for CF that are not derived from petroleum.

On this background the European Commission, in the NMP.2013.2.1-1. programme, called for research proposals to address key technological issues such as the development of new precursors, new processing routes and functionalisations for carbon fibres. The key points of the topic addressed within FIBRALSPEC project are described below:

- Technical content/scope: The use of carbon fibre based composites is of major importance in many industrial applications: transport, aerospace, electrical (Li-ion batteries, supercapacitors), pressure vessels, high cost of carbon fibres. Research is therefore needed to allow the opening of new ways for the industrial production in Europe of carbon fibres as well as their functionalisation for targeted applications, and at affordable cost.
- Proof of concept in terms of product and/or process should be delivered within the project, excluding commercially usable prototypes, but convincingly proving scalability towards industrial needs.
- Non-destructive techniques for the characterisation of fibres at the various processing steps and of functionalised fibre products may also be addressed.
- Dedicated modelling of process and properties, qualification, standardisation and/or the production of (certified) reference materials may also be addressed as an integrated part of the research proposal.
- Life Cycle Assessment (LCA) of the new processes or materials used, their energy efficiency, as well as environmental and safety issues and recycling should be addressed.

In accordance with the overall concept presented above, the project has had the following aims and objectives:

- The improvement of production of carbon fibres from "green" precursors such as lignin and renewable resources and control of precursor oxidation treatment
- The manufacturing of fibre reinforced composites via eco friendly-production techniques
- The functionalization of CFs mainly focused on cost reduction, mechanical and chemical property improvement
- The use of the reinforced composites in different applications, such as: Flexible Supercapacitors and Rapid Deployment Secure Emergency Shelters
- As for recycling and used of recycled CFs, new techniques have been used to provide commercially-
relevant products that are manufactured from waste carbon fibres

- The application of LCA to quantify the green credentials of the reuse and recycling strategies from the start -precursor development-, till the carbon fibre waste.
- Testing of laminates and prepregs production based on the new developed carbon fibres followed by manufacturing of laminates/coupons and high-performance filament wound tubes are also foreseen
- Mathematical modeling has been conducted so as to determine properties of CFs and composites, together with cost modeling

FIBRALSPEC Project is divided into 11 major Major workpackages Work Packages (WPs) (Figure 1). These constitute the main work breakdown structure of the project, gathering together the major groups of activities to be carried out. The overall structure is shown in the Figure 1 diagram above.

The main objectives of WP1 “Development of new precursors” were the improvement of production of CFs from different precursors (such as from stabilized PAN fibers, ligninocellulosic precursors, a novel polymeric structure and PAN nanofibers produced by electrospinning), the development of a resin delivery system to enable the carbon fibre CF precursors and their hybrids to be delivered to the extrusion equipment and the characterization of CFs produced by different precursors.

In addition, the major objectives of WP2 “Synthesis and functionalisation of carbon fibres – composites” included the manufacturing of fibre reinforced composites via the eco-production technique, the modification of a resin delivery unit as appropriate to enable the carbon fibre filaments to be coated by the specified resin system and enhancement of properties of carbon fibres produced.

The WP3 “Characterization of Materials and Mechanical testing” aimed to the characterization of physical properties of new fibers developed, the study of microstructure of the new carbon fibers produced and to probe the mechanical properties of the new fibers & composites developed.

While, the WP4 “Materials development (supply), Hybrid materials” purposed to develop a novel mechano-electro-spinning apparatus to the production of highly oriented precursor preforms, to develop techniques to stabilise the molecular alignment of the precursor preforms during the heat-treatment regimens and to develop novel surface multi-functionalisation technique for new fibres based on active-screen plasma. The main objective of this workpackage WP4 is to prepare advanced flexible supercapacitors based on carbon nanofibers and innovative techniques.

The WP5 “Dedicated modelling of process and properties” aimed to develop new combination of models and computational simulation method procedure based on Finite Element Method (FEM) in order to study the mechanical interaction (shearing and fracture) between fibers / matrix and to develop an integrated technique in order to study the fibre-resin interface structure and bonding and to validate the micro-mechanics mechanics model.

Furthermore, the major objectives of WP6 “Qualification, standardization, production of (certified) reference materials” were to disseminate the FIBRALSPEC project message to international experts and encourage them to apply project results to their work and explore the new markets.
In WP7 “Manufacture of Proof of concept”, the proof of concept products were excellent examples of the broad range of products that can potentially be created by the newly developed carbon fibres. It was performed the proof of concept 1 “High technology – small scale: Supercapacitors” and the proof of concept 2 “Medium technology – large scale: Rapid Deployment Secure Emergency Shelter (RDSES)”. The aim of proof of concept was to take an existing concept “Rapid Deployment Secure Emergency Shelter (RDSES)” designed and developed applying traditional composite manufacturing techniques and, by employing the use of carbon fibre to be reduced the unit mass while at the same time increasing rigidity, durability and end user usability, resulting in Rapid Deployment Secure Emergency Shelter carbon fibre (RDSES-cf). This proof of concept will can be extended to using glass and carbon hybrid structures that use the appropriate materials in the appropriate situations.

The most important objectives of WP8 “Recycling” were to specify strategies, at the start of the precursor development programme, to enable the carbon fibre waste (waste is defined as: precursor waste, carbon fibre production waste and recovered carbon fibre waste), to develop processing routes for the carbon waste generated in the FIBRALSPEC project, including those recovered from composites manufactured by the Consortium using the FIBRALSPEC carbon fibres and to evaluate the thermo-oxidative and mechanical properties of the carbon fibre composites manufactured from waste and recycled FIBRALSPEC carbon fibres.

The WP9 “Life Cycle Assessment” aimed to quantify the green credentials of the reuse/recycling strategies via LCA and LCC analyses. The objective of the “Dissemination, Exploitation and Standardization” work package (WP10) was to build consensus and raise awareness around the achievements of the whole project and how to implement them. The dissemination activities helped in achieving a critical mass of stakeholders aware of the upcoming breakthrough innovations, challenges and goals of the project. The exploitation component ensured that there was a credible plan for taking the ideas and concepts into the market and commercializing them eventually. From the FIBRALSPEC Project perspective, the successful dissemination strategy aimed:

(a) to extract and give the main messages or key implications from the project results,
(b) to identify methods to raise awareness of the project,
(c) to encourage communication between key players regarding the results of the project and
(d) to develop ways so that deliver the FIBRALSPEC project message to the targeted audience and encourage them to apply project results to their work and explore the new markets

Finally, it is referred the main general objectives of WP11 “Management”. The project had been managed according to well defined procedures aimed at the delivery of quality project results to the Commission within time and budget. The main objectives were (a) to coordinate and conduct the project according to the work plan, (b) to identify and confirm deliverables and milestones, (c) to analyze the risks associated with project execution and to take corresponding mitigation actions, (d) to ensure efficient and high-quality communication between the partners, (e) to ensure a smooth transfer of information (e.g. reports) to and from the Commission and (f) to distribute the financial support paid by the Commission among the partners.

Project Results:
A. Improvement of production of CFs from renewable resources

To date, the most common precursor used to produce carbon fibre is polyacrylonitrile (PAN). When investigating the cost of producing carbon fibre, it becomes clear that more than 50% of the cost is related to the production of the precursor. The remaining cost is distributed as such: 15% to the oxidation process, 23% to the carbonization process, and the remaining to sizing and spooling. The key to reduce the production cost of carbon fibre is the use of an alternative precursor.

Within FIBRALSPEC the production of CFs from renewable resources was extensively analyzed. Specifically, stabilization and carbonization process of rayon-based fibers and the experimental procedures to optimize the production process to a certain extent were conducted. Also the processing conditions were optimized for efficient production. The work clearly showed that by using appropriate processing conditions, it is possible to produce good quality carbon fibers from rayon precursor.

Another biopolymer with high carbon content allowing high carbon yields at low costs is lignin. If lignin is compared to PAN as precursor for carbon fibre, the ability to reduce the cost of manufacturing is by more than 50%. Lignin is a natural waste byproduct of the paper industry and biorefineries, which is readily available in enormous amounts and due to this very inexpensive. Lignin is also a sustainable, renewable resource. The use of lignin offers significant cost saving potential in the production of carbon fibre. Lignin makes it possible to produce a carbon fibre based on renewable resources.

One of the major drawbacks of lignin-based CFs is the poor mechanical properties comparing to PAN-based CFs. Blends with improved properties were fabricated via compounding lignin with suitable polymers. Despite the fact that blending process is a potential method for producing durable and spinnable materials, polar lignin powder appears low compatibility/miscibility in a nonpolar polymer matrix. For this reason, a variety of lignin modifications were conducted (acid treatment, alkylation, and esterification) in order to examine the miscibility with polymers like polyethylene oxide et al.

Another work dealing with lignin as CFs precursor is the synthesis of PAN-lignin copolymers. During copolymerization processes of lignin with specific monomers, a new renewable polymeric material is possible to be synthesized and applied as a candidate of CFs precursor. The study of bamboo as CFs precursor and carbonization/graphitization of cotton waste fibers was conducted as well.

B. Lignin Chemical stabilization

In the research field of CF production melt spinning of lignin has been an issue. Moreover, the potential of CF production and applicability of softwood lignin is under question in respect of the low mechanical properties of the resulting CFs. In our study, modification and polymer blending were conducted and optimized together with incorporation of softwood lignin in the chemical stabilization process. That showed the effectiveness of sulfonation on the introduction of thermosetting property of HDPE (hybrid high-density polyethylene)/SKL (softwood kraft lignin) fibers via chemical crosslinking, using various analytical tools.

According to the literature, no studies have been reported related to the aforementioned chemical stabilization process. This alternative route, suggested in our study, could provide a novel approach for the investigation of incorporation of softwood lignin resources, which currently remain unused, to added-value high prospect products.

During our research, stabilization of melt-spun HDPE-SKL fibers was examined involving physical (thermal) and chemical methods. Using a 3x3 factorial experimental design, sulfonation temperature and time effect on the fiber properties were examined, focusing on the thermal stability of the final product. The
optimum result in terms of morphology and crosslinking index was obtained by a two-step sulfonation process. After the sulfonation, the mass of the abovementioned fiber remained relatively stable (only about 35% mass loss) when the temperature was raised up to 900 °C and remained there for some time. Carbonization of the HDPE/SKL fiber was successful, producing a CF, which was sustained intact even at 900 °C. Regarding the morphology and the characteristics of the produced CF, they demonstrate significantly better physical properties than the precursor fibers and they are relative to similar results from recent literature. Concerning the current state of the art in this field, the present study demonstrates advantages of paramount importance. The simpler way of chemical stabilization outbalances the previous attempts on exclusively thermal stabilization of fibers, which is both much more energy and time consuming. In addition, the amount of chemical reagents used to reach this outcome is minimal. This work succeeded in investigating the possible ways of manufacturing CFs using common low cost commercial polymers and raw first materials, such as lignin. Further exploitation of this study's results can lead towards the enhancement of such production and the increase of applications in everyday life. These significant achievements show the manner in which the production of green CFs can be scaled up and overly improved.

C. Thermal treatment (stabilization, carbonization) of CFs precursors

The thermal treatment and characterization of lignin (and lignin's blends) as candidate precursors for CFs was investigated in detail. Fibres originated from lignin blends were studied by thermal oxidative treatment in various rates in order to evaluate their thermal stability. Additionally, mechanical properties prior and after thermal treatment of lignin-based fibres were measured. The results demonstrated that CFs are feasible to be produced using novel FIBRALSPEC precursors. The carbonization process can be further performed at elevated temperatures (> 1200 oC) and if graphitization (> 2000oC) could be achieved then promising CFs could be produced using the proposed method. Additionally, the application of tension during carbonization could limit the shrinkage phenomena. The serious most serious issue that should be tackled is the weight losses during high temperature process; these have to be controlled in order to improve the yield of the process. This aim could be achieved by dehydration of the fibres prior to their thermal oxidative treatment, preferably by chemical means (e.g. by their impregnation with flame retardants, which enhance thermal stability and prevent the evolution of volatile by-products by blocking the primary hydroxyl groups).

In case of thermal treatment for lignin, the modification by acid resulted in more homogeneous and more thermally stable material. Moreover, the alkylation by bromododecane increased the initial decomposition temperature and the esterification by phthalic anhydride showed the wider decomposition region (about 300 oC), but otherwise caused decrease of the thermal stability of lignin. In the end, the esterification by maleic anhydride leaded to narrow enhancement of thermal stability.

D. Continuous stabilization line (for oxidation treatment of CFs precursors)

One of the main tasks was on improving the most crucial step of the process, i.e. the oxidative stabilization. The stabilization and carbonization processes of PAN-based fibers and the experimental procedures to optimize the production process to a certain extent have investigated. The specific objectives were to evaluate the stabilization stage with respect to mechanical properties of the PAN-based fibers. The aim of the study was to establish the rules for designing oxidative thermal treatment that led to the optimization of fibre stabilization, using the results of their isothermal treatment. The optimal PAN fibre
stabilization conditions were achieved when the effect of the decomposition reactions was minimized. A simple experimental protocol, which can be followed and applied not only in laboratory but also in larger scale, has been provided, after the examination of optimization of PAN oxidative stabilization and subsequent pyrolysis. NTUA has designed and established a pilot unit (Figure 2) in order to produce stabilized lignin fibre at high volume. Automatic control system of stabilization line can optimize the aforementioned procedure by changing a variety of parameters.

Within FIBRALPSEC the sequence of CF production process from green fibres as described below was followed:

Stabilization. During stabilization of the fibre; dehydration, rearrangement, formation of carbonyl groups, evolution of carbon monoxide and carbon dioxide, and formation of carbonaceous residue occur alongside the thermal scission of glycosidic bonds between the glycopyranose units of the cellulose. The scission produces oxygenated compounds that lead to maximum loss of the mass. Tars and coke residue are the main volatiles gaseous products formed during the thermal degradation. By adding proper chemical such as impregnants or flame-retardants, one can moderate the stabilization mechanism by lowering the temperature range and subsequently improving the carbon yield by reducing the burning loss as well as having better stabilized fibres for further processing (carbonization and graphitization).

Carbonization. This step includes heating of the fibres between 500°C to 1500°C (high temperature processing). During this step, non-carbon atoms are removed from the atomic structure. At this stage, all chemical processes are completed, structural transformations take place, the coke becomes enriched with carbon, and the fibre properties alter. The resulting fibres have bonded crystalline structures with better alignment and orientation. As the temperature rises, the structure of the residue becomes more complicated, in spite of simplification of the elemental composition.

Graphitization. It is the final stage of the production process (higher temperature processing). Most of the process is energy intensive and is responsible for the high cost of CFs. Depending on the fields of the final application, either carbonized or graphitized CF can be the end product. The main phenomena occur are structural transformations and simultaneous changes in the properties of the fibres. The most important processes during this stage are: further aromatization of carbon atoms, growth of crystallite size and graphite like ribbons, and perfection of the orientation of the ribbons along the fibre axis.

E. Screening Study
Selection criteria have been proposed to evaluate cellulose, lignin and other natural polymers as precursors for manufacturing carbon fibres. A template to enable short-listing of potential precursors, and the subsequent development of a technical document, was issued to the WP1 partners. The key attributes implied in the template were as follows:

(a) Precursor Related Costs – the availability (scarcity) and cost of the precursor, cost associated with any secondary/tertiary chemicals and processing; recycling of solvents; separation of precursors from by-products; purification of precursors; disposal of primary waste; pre-preparation costs prior to fibre formation (drying, etc); flushing/cleaning materials cost; disposal of secondary waste.

(b) Preform and Fibre Production Costs - local ventilation and extraction (LEV); costs associated with the primary and secondary processing equipment for producing preforms and fibres. This will be based on the specialist equipment being developed / used by the relevant partners including items required for melt-extrusion, wet-spinning, dry-spinning and mechano-electro-spinning.
(c) Energy and Utility Costs for Preform Production, Stabilisation, Carbonisation and Graphitisation – it is envisaged that the equipment costs in this category was covered by item (b) above. The current category is linked to the cost of power (electricity, gas, etc) and utility (water, inert gas, compressed air, etc).

(d) Properties of the Fibralspec Carbon Fibres – this is proposed as the primary criterion for evaluating the fit-for-purpose of the carbon fibres produced in the project.

(e) Environmental Impact – this is not covered explicitly during screening as it is the subject of WPs 8 and 9.

F. Development of a novel synthetic polymer as possible new CF precursor

One of the main objectives was to develop a novel synthetic polymer as possible new CF precursor. In order to improve different aspects of CF production, a different approach was applied: developing novel tailored PAN block copolymers (as a means for improving quality of CF). Initial research results demonstrated that it is possible to synthesize acrylonitrile-based copolymers with specified structure using activator generated by electron transfer atom transfer radical polymerization (AGET-ATRP) polymerization method in microemulsion system. The new polymer was designed with a structure containing aromatic structures. This material is thought to be a good candidate for CFs since it demonstrates large carbon yield after pyrolysis (increasing the process efficiency) and could possible modified to CFs with enhanced properties. According to the synthetic process, at first, the synthesis of the proper monomer is performed taking into account that it should have enhanced reactivity (react in mild conditions) and absence of solvents during polymerization. It is mentioned that special attention is given to the physicochemical properties of the fabricated polymer (especially its thermo mechanical behaviour), in order to propose suitable production methods. After the fabrication of the new fiber precursors, the experimental parameters for their modification into CFs were investigated and corresponding protocols were developed related with the pyrolysis as well as stabilization processes. The benefits of the aforementioned study are considered to be: a. fulfillment of monomer production expectations, b. new cost-effective material for utilization in CFs industry.

G. Fiber spinning

Melt spinning process has been proven by far that is better spinning process than dry spinning in several aspects. Melt spinning apparatus is easily handled, easily cleaned, can run with a small amount of material in the form of powder or pellets (>5g) and achieved continuous production of lignin-based fiber. On the other hand, dry spinning did not manage to produce an acceptable length of continuous fiber. Moreover, the organic solvents used in spinning dope solutions are toxic and dangerous for human health. Continuous production using commercial polymers was achieved via melt spinning. It is observed that unmodified lignin forms agglomerates in the extruder when it is mixed with commercial polymers, resulting on larger fiber diameters due to decreased compatibility between materials. This problem was solved by modifying lignin and resulted on smaller fiber diameters and better compatibility with commercial polymers, such as Polylactic acid (PLA) and Polypropylene (PP).

Finally, it was noticed that chemical modifications of lignin increased the molecular weight, enhanced compound miscibility and improved the produced material’s thermal stability.

Work Package 2: Synthesis and functionalization of carbon fibres – composites

For today, carbon-based materials have received much attention for their many potential applications. The
carbon fibers are very strong, stiff, and lightweight, enabling the carbon materials to deliver improved performance in several applications such as aerospace, sports, automotive, wind energy, oil and gas, infrastructure, defense, and semiconductors. However, the use of carbon fibers in cost-sensitive, high-volume industrial applications is limited because of their relatively high costs. However, its production is expected to increase because of its widespread use in high-volume industrial applications; therefore, the methods used for manufacturing carbon fibers and carbon fiber-reinforced composites and their structures and characteristics need to be investigated.

One of the most actual tasks of all researchers interested in carbon fibers and polymer based composites reinforced by carbon fibers is directed changing of their structure, physical and chemical properties for enhancing of their service characteristics and opening of new functional possibilities of their using. Carbon fibers have received much attention lately for their many potential applications in different matrix materials owing to their properties, processability, and recyclability. However, raw CFs need to be treated and/or sized as part of the manufacturing process. Designing of suitable surface-treatment method is a requisite to ensure that the high strength of the CFs is maintained during handling and composite manufacture. The surface treatment or sizing method is also equally important to ensure the formation of CF-matrix interface. In the process of creation of polymer based composite materials the special attention should be paid to the study and control of the interactions at inter phase boundaries. It is very important for the composite materials on the base of various kinds of polymers reinforced by the high modulus carbon fibers.

A. Surface treatment (electrochemical, plasma, nanoprofiling and nanomodification) of CFs

1) The aim of this work was to enhance the adhesion strength between the fibers surface and the polymer matrix by either producing oxygen active group at the surface of the carbon fiber by mean of electrochemical treatment or by grafting the fiber with polymer coatings with either carboxyl groups or aromatic rings. 

The electrochemical treatment was conducted using cyclic voltametry conditions whereas the electropolymerizations were conducted using potensiostatic conditions. The resulting fibers were studied using methylene blue discoloration, XPS, SEM, IR, and contact angle measurements. The coated fibers have been quantitatively (SEM) and qualitatively (FT-IR, XPS, Raman and contact angle measurements) studied demonstrating the electropolymerization onto the CF’s surfaces. The IR and μ-Raman spectra of the coated fibers exhibited the characteristic peaks of each polymer which stand as proof for the successful electrografting of the fibers. In addition, SEM images depicted the electrografting of polythiophene-co-polyphenylene which produced a thick layer whereas the polyacrylic acid generated a rigid but not so homogenous polymeric layer. To further investigate the results regarding the electropolymerization TGA testing of the coated fibers has conducted in order to estimate the weight percentage of the coated polymer as well as the thermal behavior of the coated fiber.

2) Commercial carbon fibres, as well as FIBRALPSEC carbon fibres, were treated by reduced pressure plasma treatment using oxygen as process, or by atmospheric pressure plasma treatment in open air. Low pressure plasma was preferred for most treatments, due to the possibility to use oxygen gas, which was not available for atmospheric pressure treatments. All the fibres were characterized, after plasma treatment, against their morphological and compositional properties by means of FESEM and EDS respectively. The surface chemical bonds were investigated by means of XPS, exploiting survey spectra as well as high resolution spectra. The measurement of the plasma parameters in an active screen setup
using a triple Langmuir probe provided important evidence to explain the formation of the active species responsible for the surface functionalisation of the carbon fibres. These results will help to optimise the treatment conditions as well as to scale-up the plasma reactor when required. The tests to determine the effect of plasma treatments on the bond strength of CFs in composites were executed as well.

3) The technology regimes for production of the following carbon containing nanoparticles from plant raw materials had been proposed: amorphous carbon, nanographite, silicon carbide and graphene nanoparticles. Based on the nanoparticles characterization the following particles were selected: graphenes in the form of aqueous solution recommended for CF surface nanoprofiling; other kinds of nanoparticles (amorphous carbon, nanographite, silicon carbide) had been recommended for nanomodification of coupling agent on the base of epoxy resin.

Experiments on preliminary treatment of CFs surface before nanoprofiling had been studied. The most effective method is annealing in air at the temperature 350 °C and oxidation in concentrated solutions of sulfur acid and hydrogen peroxide. Nanoprofiling by the solution of graphenes revealed the best results after oxidation annealing at 350 °C and chemical treatment by concentrated solutions of sulfur acid and hydrogen peroxide. Tensile strength of CFs after graphene nanoprofiling increased up to 10-25 % in comparison with initial value.

The technology of nanomodification for coupling agent from epoxy resin by means of the nanoparticles of amorphous carbon, of silicon carbide and nanographite had been developed. Microplastics containing epoxy resins modified by carbon nanoparticles (about 0,5 %) had been produced. It was concluded that the strength of micro plastics with nanomodified coupling agent increased up to 10%-15% in comparison with initial fibers. The main advantages of graphene coatings produced from aqueous solutions are effective defect healing (defects of CF surface) and increasing of CF strength. Furthermore graphenes are well wetted by epoxy binder and it creates the conditions of effective using of CF with graphene coatings in composites manufactured by prepreg method. Important technological advantage of graphene solutions is their relatively long life time from the moment of their production till the moment of their using for creation of coatings on the surface of carbon fibers(not less than one month).

Aqueous base of these coatings creates better ecological conditions for the operations of creation of coatings on the surface of carbon fibers. Dry nanoparticles of graphite and amorphous carbon are very effective for nanomodification of epoxy based binder. Nanographite particles to be induced in epoxy binder essentially strengthen interface boundary fiber-binder by increasing of the number of supramolecular bonds and increase the composite strength in whole. The detailed information concerning the study of tensile strength of nanomodified microplastics reinforced by carbon fibers and positive influence of nanoparticles on mechanical properties of microplastics is presented as well.

Surface treatment techniques that were applied within FIBRALSPEC significantly affect the CF properties. These treatments on the surface contributed to enhancing the interfacial bonding between the fibers and the matrix in the composite that leads to enhance the friction co-efficient and hardness significantly. Almost two folds of mechanical enhancement were observed in composites. This effect may be due to increase of the fiber-matrix interfacial adhesion, which is enhanced with surface-treatment effectiveness on carbon fibers. In addition, carbon fibre roughness plays important roll. The increase in composites hardness is indication of good bonding between the matrix and the reinforcement materials. The mechanical behaviour indicate that appropriate use of surface modified carbon fiber in composite processing may contribute to enhancing the interfacial and/or inter laminar properties of carbon fiber reinforced composites, depending on their resin characteristics and processing parameters.
B. Technical performance of CF composites
Unidirectional (UD) composites were manufactured via “the clean filament winding”. Impregnation during the clean filament winding was influenced by the permeability of fibre configuration, porosity or fibre volume fraction, resin viscosity and the fibre dimension. The degree of impregnation is calculated, which is important for determining the winding speed.
Two types of carbon fibres were used in manufacturing the UD composites, namely T700S and NTUA fibres. Both the fibres were characterised by conducting density measurement and FTIR analysis. The density of NTUA fibres is lower than that of T700S.
The physical and mechanical properties of the UD composites manufactured in UoB were then evaluated. The tensile and interlaminar shear stress properties of the UD composites reinforced by the NTUA carbon fibres were lower than those of the composites reinforced by commercial T700S carbon fibres. The relatively low interlaminar shear stress value implies that the bonding between the NTUA carbon fibres and the epoxy matrix could be improved further by surface modification/functionalisation to achieve optimal performance.

C. Production of textiles
Knitted fabric used as a filler for composites is another promising technological material that was studied within FIBRALSPEC. In comparison to traditionally used textile materials, knitted fabric has a list of advantages. Thanks to stitches structure and high deformation properties knitted fabric has high molding properties that allow simplifying the production of complex shape products. Knitted structures build by interweaving with one or several reinforcing yarns allow realisation of more wide range of shapes and properties than woven. Structure of knitted interweaving has bigger variety and allow significantly change the knitted fabric parameters effecting on its properties and allow to receive both low stretchable knitted fabric which deformation is comparable to cloth deformation, and high stretchable knitted fabric which extensibility is 150-250%. Quite important are advantages of knitted method manufacturing: high productive capability of knitted equipment, reduction of technological processes number, versatility of knitting machines during processing of different type of raw materials, high computerization in production of knitted products. Therefore, in certain cases, knitted fabric is irreplaceable reinforcing element for composite materials, and its production from economical point of view is cost effective. Knitted structures provide fabric deformation in all directions and applicable for production of composite products by mold forming method. However, such knitted structures are not applicable for high modulus carbon fibres that are the most perspective fibres for high performance applications.
The major challenge was development the interweaving methodology of knitted structures from high modulus carbon fibres that have low level of physical and mechanical properties. Targeted goal was achieved by selection from the variety of knitting interweaving types for high strength and high modulus carbon fibres of such interweaving types that will not require looping, i.e. no yarn bending by knitting machine needles, but introduction of direct yarn in fabric. In order to use high modulus carbon fibres on rib machine, carbon weft knitted fabric was produced where high modulus fibre (fragile) is used as weft, and low modulus CF is used as warp, for example, based on cellulose hydrate.

D. Handling and post-processing of CFs
The procedure for handling and post-processing carbon fibres was presented in detail as well:
1) Surface treatment of carbon fibres
2) Preform manufacturing, which carbon fibre bundles are woven or knitted into intermediate reinforced products (often called as preform)
3) Tensile properties of single fibre and fibre bundle

Work Package 3: Characterization of Materials and Mechanical testing

Within FIBRALSPEC a wide range of characterization methodologies was applied in order to identify promising materials and processing conditions to produce new CFs and carbon nanofibres for specific applications: composite materials and supercapacitors. The work conducted in this part of the project included:

• The adoption of suitable procedures for handling the fibres and composite materials, in order to obtain reliable and repeatable results with the measuring and testing techniques.
• The selection of appropriate methodologies and parameters for the characterisation of carbon fibres with Field Emission Scanning Electron Microscopy (FE-SEM), Energy Dispersive X-Ray Spectroscopy (EDS), Transmission electron microscopy (TEM), Atomic Force Microscope (AFM), X-ray photoelectron spectroscopy (XPS), X-ray Powder Diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), Raman spectroscopy, to determine the chemical composition, the internal structure and the surface morphology of commercial carbon fibres (benchmarks).
• The application of the methodologies to commercial carbon fibres and composites (benchmarks) and new materials developed for the project.
• The extraction of conclusions to help identify promising materials and processing conditions to produce new CFs, carbon nanofibres and composite materials for particular applications.

The work conducted by the partners of WP3, in relation with the determination of physical properties of carbon fibres, nanofibres and composite materials, is summarised in the following key points:

• A methodology was established for the accurate determination of the diameter of carbon fibres and precursor fibres. These measurements are important for the subsequent determination of the tensile strength, and other mechanical properties, of single filaments.
• The thermal stability of fully graphitised carbon fibres was studied in air and inert gas, providing a reference point for subsequent studies of new precursor fibres (rayon and lignin). This information is valuable for the determination of the heat treatment parameters used for the stabilisation and the carbonisation processes.
• Contact angle measurements were conducted on single carbon fibre filaments and on vitreous carbon discs, as proxies for carbon fibres. The measurements revealed differences in wettability as a function of the applied surface treatment, namely cyclic voltammetry and plasma. The measurements are relevant for the interfacial bond strength between carbon fibres and polymers in composite materials.
• The dimensions of carbon nanofibres, carbon nanotubes and carbon nanoparticles were determined by high resolution SEM and TEM. This information is valuable for the subsequent use of such nanomaterials in composites and/or in supercapacitors.
• A methodology was developed to measure the electrical resistivity/conductivity of carbon nanomaterials. The methodology provided useful insights into the electrical properties and potential applications of the carbon nanomaterials used in this project.
Mechanical properties are the most important service characteristics as for carbon fibres (yarns and monofilaments) and fabrics on the base of them and also for microplastics and separate prepregs and composites reinforced by carbon fibres and carbon fabrics.

Within FIBRALSPEC the mechanical and physical properties of carbon fibres i.e. commercial carbon fibre as benchmark and the newly developed carbon fibres in the project such as NTUA fibre, Polito cotton fibre and UOB lignin fibre were investigated. The mechanical properties about mono-carbon fibres include tensile strength, yield strength obtained through tension test, and modulus and nano-hardness via nano-indentation etc.

Micro-plastics is a model plastics that consists of carbon fibres impregnated with the binder and arranged in one or two layers followed by solidification. It is a convenient way for investigating the influence of plasma treatment on the carbon fibres and their composites. Micro-plastics based on initial fibres and plasma treated fibres have been fabricated in IPMS and SDO Yuzhnoye and their mechanic properties like tensile strength have been evaluated. Composites based on modified/untreated carbon fibres and epoxy binder fabricated in Polito, IPMS, UOB and NTUA have been introduced and their mechanical properties like hardness (Nano- and Micro), tensile strength, yield strength, shear strength, flexural strength and scratch resistance have been measured.

A. Carbon fiber/Matrix Interface

The mechanical properties of CFs are strongly linked to their microstructure and, in turn, the microstructure is a function of the precursor materials and the processing conditions. The interface between the fibre and the matrix plays an important role in the mechanical properties of fibre-reinforced composites i.e. the fracture mechanism and the fracture toughness, which often influences significantly the composite performance in all types of composites. The interfacial strength of the carbon fibre and resin has been assessed by push-in and push-out test via a nano-indentation techniques, and the creep behaviour of the single fibre in a carbon composite is also reported.

The knowledge gained on the properties allows to perform detailed analysis of various classes of materials based on carbon fibres for different applications, to formulate the recommendations concerning the directed changing of service parameters to choose the ways of enhancing of such kind of parameters taking into account the environment specialties. Emphasis was place on the interface between the fibre and the matrix plays an important role in the mechanical properties of fiber-reinforced composites and often influences significantly the composite performance in all types of composites. The interfacial properties also play an important role in the fracture mechanism and the fracture toughness of composites. Interface debonding is an important mechanism of energy absorption during the failure of a composite interface. So the experiments in tension and compression modes and their interpretation in micro and nanoscale had been performed. And fractography and microstructure studies were the main instruments that allowed to understand the nature of properties formation and to establish the criteria of directed changing of such properties and creation of composites with given complex of service parameters.

The main objects of the investigation were the single carbon fibers (monofilaments) carbon fibre yarns, microplastics and composites reinforced by carbon fibers of various brands.

The results of experiments on mechanical properties performed in the framework of the project allows to perform the detailed analysis of various classes of materials based on carbon fibers for different applications, to formulate the recommendations concerning the directed changing of service parameters to choose the ways of enhancing of such kind of parameters taking into account the environment specialties. Furthermore, the main results of mechanical properties investigations have a special practical application.
within the framework of FIBRALSPEC project. They create the base of designing of special facilities (working places) for the pilot line on composite production at SDO Yuzhnoye and IPMS. The most effective kinds of thermal and chemical treatments and the detailed parameters of such technological processes had been used for designing and manufacturing of the corresponding equipment for several working places. Instrumented nanoindentation is a valuable tool to evaluate both the mechanical and the time-dependent properties of the carbon fibre composites. The push-out tests were carried out at different testing conditions in terms of loading rate, peak load, hold time, atmosphere (different humidities) and temperature (room temperature, 50, 80 and 100°C). Based on the experimental results, the following conclusions can be drawn:

• Both the Berkovich and Cone indenter have been used for push-in/push-out or creep test and the later has some advantage due to its extended range of displacement before interference with the surrounding resin.
• A single fibre needs a critical load/stress to be pushed out under a continuous increasing load mode, and the critical load is strongly linked to the thickness, the location of the fibre in the resin and other environmental factors like temperature.
• Carbon fibres are also susceptible to creep at a load lower than the critical load, and the displacement/creep rate is closely relating to the loading conditions. Generally, the displacement distance increases with the increase of the peak load and the holding time at the peak load. However effect of the loading rate is complex and it is also dependent on the shape of diamond tip used. A fibre in a thin composite disc has a large displacement under the same load conditions.
• A fibre can be push-out during the dwell time at a stress/load lower than the critical load which is also influenced by the loading and environmental conditions.
• It was found that the fibre becomes difficult to be push-out at elevated temperature and the displacement and creep rate are both reduced with the increasing of temperature due to the resistance caused by the expansion of the fibre and resin.

Further analyse on the creep parameter A/d(0), creep strain rate and the creep compliance is helpful to quantitatively assess the viscoelastic/viscoplastic response of the fibre composite systems.

Work Package 4: Materials development (supply), Hybrid materials

Within WP4 of FIBRALSPEC project, (a) plasma surface functionalisation of CFs and of carbon nanofibres (CNFs) has been conducted, (b) prototypes of advanced flexible supercapacitors were fabricated and (c) novel mechano-electro-spinning apparatus was developed.

A) Plasma surface functionalisation of carbon fibres (CFs) and of carbon nanofibres (CNFs)
Carbon fibres (CFs) are one of the most effective reinforcements for polymer matrix composites. The properties of carbon fibre reinforced polymers (CFRP) depend, to a large extent, on the interaction between the strong CFs and the soft polymer matrix, most frequently an epoxy resin. Plasma surface functionalisation of carbon fibres (CFs) and of carbon nanofibres (CNFs) has been conducted. The results show that N2-H2 plasmas can effectively modify the surface chemistry and properties of the CFs, making it more compatible with the polymer matrix. The ageing of the plasma functionalised carbon fibre surfaces over time was also evaluated. Cotton-based carbon fibres produced from waste fabric have been functionalised by short plasma treatment with oxygen, and an increase in C-O, C=O and O-C=O bonds while the decrease in surface π-π* bonds were observed due to oxygen plasma radicals.
Plasma diagnostic studies were also carried out using two techniques: optical fibre assisted optical emission spectroscopy (OES) and triple Langmuir electrostatic probe, to assist in optimising the plasma condition in an innovative active-screen plasma (ASP) surface engineering technology. A relationship was found between the active species in the plasma and the functional groups attached to the carbon surfaces, which provides valuable information for the optimisation of the ASP treatment.

In summary, a new active screen plasma technology has been developed to functionalise the surfaces of carbon fibres and carbon nanofibres respectively by ASP modification and deposition of Ni and Ag nanoparticles. Significantly improved surface properties have been successfully achieved by the new ASP surface functionalisation technology.

B) Prototypes of advanced flexible supercapacitors

A prototype of a supercapacitor (Figure 3) fabricated was developed, using electrodes based on mixtures of carbon nanofibers and graphene related nanomaterials. The work on the fabrication of the prototype has consisted in different steps. Firstly, the determination of the specific concentration of Graphene and CNFs mixtures to achieve the best results was performed. Secondly, the devices were physically fabricated. Finally, the devices which have characteristics that match quite well the objectives defined were tested. To reach this objective, the physical-chemical characteristics of the nanomaterials, mixtures achieved (that constitute the electrode) were evaluated and the electrochemical tests on the electrodes/cells were performed.

After fabricating CNFs based electrodes, different physical characterization was performed. SEM images were obtained in order to evaluate the intercalation of CNFs between the graphene layer, BET measurements to evaluate the potential surface that could be exploited, Auger measurements to understand and find solutions to the oxidation of the aluminum based substrates/collectors that can increase the total resistance of the supercapacitors and so reduce the power delivered.

The electrochemical characterization of the electrodes is an essential step in the project. After fabricating the electrodes and performing the physical characterization of the electrodes, the electrochemical characterization of the electrodes and cells fabricated was performed using specific electrolytes. Through this phase, the real added-value of the nanomaterials in fabricating supercapacitors electrodes was understood and evaluated.

After these measurements, the prototype with a surface of 10x10cm² (a whole cell with electrolyte inside) has been fabricated (with aluminium as collector). The final prototype to a device composed by 10% of CNFs and 90% of rGO, that allows to achieve 20F/g as capacitance and 40kW/Kg using ionic electrolytes on aluminium substrate using oxygen-free solvents to avoid oxidation. A prototype exploiting the same concentration and electrolyte but with graphite as collector and using water based suspensions was also fabricated in order to be evaluated the widening of the interval exploiting graphite considering that interaction of ionic liquid with the aluminium for the larger values was observed. The dimensions of the devices are 10cmx10cm packaged.

C) Novel mechano-electro-spinning apparatus

A novel Mechano-Electro-Spinner (MES) has been designed, constructed and commissioned (Figure 4) at UoB in collaboration with CTM. CTM has completed the MES, particularly related to the high voltage electricity and a manual with the Operation and Maintenance of the MES was prepared. The Mechano-Electro Spinner has been designed to be versatile for Laboratory trials in the manufacture of
precursor fibres intended for the production of carbon fibres. There is a 27.5 cm$^3$ capacity cylinder designed to melt solid materials which are displaced by virtue of piston connected to a linear axis, the now liquid is forced through chosen diameter dies or needles. This extruded material can be post manipulated by a variable combination of in line rollers, heaters and coolers. The extrudate is then collected on a rotating cylinder designed to stretch and collect in a controlled way. Combined with the mechanical manipulation of the extrudate is the ability to add high voltage, traversing a short distance from the needle through the extrudate to the collector drum.

The Mechano/Electro Spinner is housed in an enclosed translucent polycarbonate and aluminium frame. This frame has two interlocked doors to restrict access to the machine while running in automated modes. Manual access is granted via the doors in order for manual intervention to assist with starting and stopping the process. A full colour operator touch screen (HMI) is situated on a moveable arm. The HMI allows control and adjustment of the control and use of the equipment. It allows access to different running modes which enables/disables certain features in line with the operator intervention. It is imperative the machine is used in accordance to the safety manual / documentation in order to prevent damage or harm by its misuse.

Work Package 5: Dedicated modeling of process and properties

Within WP5 framework, the development of experimental methods, computational tools and modelling activity for characterisation of carbon fibres/matrix interface, namely i) modelling of stabilization process, ii) chemical iii) and micromechanical interaction between carbon fiber and matrix, was carried out. The work conducted is summarized and the key results are highlighted as follows:

1. The selection of appropriate methodologies and parameters for the characterisation of interfacial properties by micromechanical techniques such as push-in/push-out tests.
2. Push-in/grid nanoindentation protocol (Figure 5) was applied on samples prepared from T700S composite at room temperature. The varying parameters of the grid nanoindentation were the orientation of the CFs with respect to the indentation axis and the local microstructure (local volume fraction of the CFs, CFs packaging). The morphology of the surface was examined by SPM in order to avoid damaged surface regions. After performing each grid indentation SEM and SPM were combined in order to be matched nanoindentation imprints with load-displacement curves.
3. Push-out tests were conducted on thin sections of a T700S composite at room temperature, high temperature (125°C) and high relative humidity (90% HR) to assess the micro-mechanical interaction between the carbon fibres and the polymeric matrix. The SEM and AFM observations confirmed the push-out of individual fibres up to a distance in the order of 1.2 μm. The analysis of the load-displacement curves revealed higher compliance at high temperature and lower compliance at high humidity.
4. The testing methodology consisting of environmental push-out tests produced valuable information, but a large number of tests are required in order to obtain statistically valid results in view of the large number of independent variables of the system.
5. The results obtained from push-in/push-out tests under various experimental conditions (loading rates, maximum load, dwell time, thickness) provide valuable information regarding deformation mechanism and failure modes of CF/matrix interface.
6. A 3-D numerical model was developed utilizing a cohesive zone model (CZM) based on the commercial FE software ANSYS 16.0 to simulate the progressive failure of the fiber-matrix interface during push-in/out
7. The effect of mesh density in numerical results was studied and corresponding analysis was performed. The effect of various parameters such as friction coefficient (fiber/matrix), mechanical properties of the matrix and interface (fracture toughness, shear strength) on the load-displacement curves of the model were calculated and compared. The distribution of Von Mises stress in the fiber-matrix interface is given for various indenter displacements into fiber and for different friction coefficient.

8. A suitable model for process design of polyacrylonitrile (PAN) fibres oxidative stabilization is established, with utilization of length change. Such model is based on parameters of time, temperature, stress, and provides a simple way to manipulate PAN stabilization without the need of in depth analysis of stabilization reactions.

The micro-mechanical interaction between the carbon fibres and the polymeric matrix in composite materials can be assessed by push-in/grid nanoindentation and push-out tests under room conditions as well as environmental conditions, namely elevated temperature and high relative humidity (90% RH). The dislodgement of individual carbon fibres up to a distance in the order of 1.2 μm was found to be suitable for the Berkovich indenter. Larger displacements resulted in contact between the Berkovich indenter and the surrounding material, thus invalidating the analysis of the load-displacement curve. A specially made cone indenter allows a large displacement about 2.4 μm thus giving a more accurate result.

The samples tested at high temperature (above Tg) exhibited higher compliance, whereas the samples tested at high relative humidity (90% RH) showed a stiffer response. This was attributed to the relaxation of residual stresses from the curing process during the high temperature experiments, and the increase in the radial compressive stress on the carbon fibres in the experiments conducted at high humidity, because of swelling of the polymeric matrix.

In addition, the results obtained using different loading rates and the sudden failure of the fibres observed during the dwell time at different constant load are indications of a time dependent response. This phenomenon is consistent with the SEM observations of the push-out fibres, and with the viscoelastic properties of the polymeric matrix reported in the literature for macroscopic tests. Therefore, the viscoelastic failure theory appears to be more appropriate to model the push-out tests.

The environmental push-out tests provided valuable information to characterise the micro-mechanical interaction between carbon fibres and polymeric matrix in composite materials. However, a large number of tests are required in order to obtain statistically valid results, in view of the large number of independent variables involved in the system.

The nanoindentation multicycle grid methodology in the push-in test was integrated into an experimental protocol combining SPM and SEM imaging for the detection of differences on the indentation induced failure behavior of a commercial CF reinforced epoxy matrix composite as a function of CF orientation and local microstructure. Pop-in discontinuities were mainly produced by CF cracking and they were present only at 0° and 45° samples. At the 0° and 45° samples the one third and one sixth, respectively, of the indentations produced pop-ins. It was observed that the larger the matrix pocket area, the higher the pop-in load, due to the fact that the matrix is more compliant with respect to CFs. Morphological observations on the indented areas revealed different failure behavior of the CFs, namely shattering and distinct fracturing, depending on their orientation with respect to the surface. Measured reduced elastic modulus and hardness values were lower after the occurrence of pop-in.

Furthermore, a computational scheme based on Finite Element Method (FEM) has been developed to elucidate the deformation mechanism of interface during push-out test. This is facilitated by the database of the mechanical properties of the studied materials produced within WP3 by mechanical tests such as
nanoindentation, tensile etc. The main steps for the construction of the computational model on a commercial software are defined, namely: (1) representation of push-out geometry, (2) definition of materials properties, (3) application of the boundary conditions (BCs) and contact type models ((i) contact-target elements and (ii) cohesive zone model for fiber/matrix interface) and (4) selection of discretization in the fiber-matrix interface. Within this framework, FE simulations were performed and post processed results were used in order to validate the model. The geometry of the push-out model was built based on experimental set ups. Since, push-out experiments were conducted on samples with various thicknesses, the parameter of thickness was studied in the simulations. It was observed that the thickness of samples (varying between 30 and 70 μm) does not significantly affect the response of load – displacement curves and the maximum load of simulation results is comparable with experimental results.

The methodology for the utilization of the carbon fiber precursor length change during the oxidative stabilization as process control parameter has been developed. In order to achieve this, a model is proposed that can describe the fiber length change in the entire process treatment window; the fundamental idea of the model is the assumption that the change of length is the sum of the contribution from the three main components, namely entropic shrinkage, creep and chemical shrinkage. By the proposed approach the contribution of each phenomenon that take place is deconvoluted. The model is applied for predicting the yield of the nitrile cyclization and the results are used to further investigate the reactions during the stabilization of carbon fiber precursors. UoB reported hyphenated techniques to monitor the curing reaction of epoxy matrix at similar process condition. The hyphenated techniques are a combination of differential scanning calorimetry DSC, Fourier Transform Infrared FTIR, and Fresnel reflection sensors.

The chemical interaction between carbon fibres and matrices was investigated by using differential scanning calorimetry DSC. For the practical reason, carbon fibres must be milled into powder. Some exothermic energies released during the curing of epoxy were absorbed by carbon powder. The decrease in heat flow due to the carbon powder in epoxy is still being investigated. However, the curing kinetics of epoxy was not strongly influenced by the carbon powder.

In conclusion, the modeling approaches developed within WP5 contribute to the theoretical study of carbon fiber integrity (model of stabilization) and carbon fiber composite integrity (chemical and mechanical interaction between carbon fiber and matrix).

Work Package 6: Qualification, standardization, production of (certified) reference materials

The aim of WP 6 was to tail developments in CF technologies, products and research trends globally and in the practical point to follow up through a common template materials flow and handling throughout the project. Standardisation is a key aspect for FIBRALSPEC and the materials monitoring was the first step towards this direction. The objective of WP6 was to establish a typical approach to follow up improvements in materials and production methodologies suggested through FIBRALSPEC in order to monitor strain, temperature, vibration, acoustic emission, cross-linking chemistry, carbon fibre content, electrical conductivity, displacement and mechanical properties. The intervention was to follow up systematically progress and improvements. Activities were to implement existing standards associated with characterisation and testing activities related with the project and identify gaps in the body of standards associated with areas of interest for the project.

Based on standards ASTM D 3379-75 “Standard Test Method for Tensile Strength and Young’s Modulus of fibers” and ASTM C 1557-03 “Standard Test Method for Tensile Strength and Young’s Modulus for
a draft methodology was developed for samples manufacturing to determine physical and mechanical properties using single fibre (filament).

Several existing standards, associated with the activities of the project, were identified and their implementation has been proposed. A gap in the body of standards was identified, particularly associated with the push-out tests used for the determination of the bond strength between the carbon fibres and the resin (WP5). The experimental work is documented and could potentially be used as a basis for a new standard.

In addition, the activities of the ISO technical committees of relevance to the Fibralspec project have been followed. With this regard, the development of new standards and the update of existing standards are of particular interest.

Finally, opportunities for development of new standards have been identified in the area of mechanical testing, namely: push-out/in tests of composite materials and compression tests of single carbon fibres (single filaments). In both cases, the testing methodologies are being carefully documented for their potential implementation as standards in the future.

Within FIBRALSPEC project, a new standard, which is being drafted by the ISO technical committee TC61 is identified. That is “ISO/CD 20975-2 Carbon-fibre-reinforced plastics — Methods for measurement of through-thickness laminate properties — Part 2: Considering size effects by flexural test.”

The future prospect uses of carbon fibers can be summarized as:

- Aerospace growth climbs with carbon fiber
- Carbon fiber quadraxial fabric has been created and investigated
- Opportunities in orthopedics are seen already in the market
- Carbon fiber has a niche, and it tries to secure a larger piece of a high-growth pie
- Initiation of use in construction projects is seen for various uses.

News relevant to CFs the last period include:

- growing use of carbon fibre by the automotive industry - Micro Hybrid Vehicles
- new carbon fibre applications in marina, buildings and infrastructure
- innovative new precursor materials targeting lower production cost
- rapid curing of composites
- Mitsubishi Rayon’s carbon fiber sheet molding compound (SMC) has been adopted for the rear door frame of the new Prius PHV
- researchers 3D print aerospace-grade carbon fiber
- CSIRO tech could increase speed of carbon fiber production by wet spinning
- styrene-free prepreg formulations with longer room temperature shelf life, shorter cycle times, and reduced cost in order to increase productivity, decrease scrap and material costs, and enable adoption into the automotive industry
- improved carbon fiber for thermoplastic applications
- composite overwrapped pressure vessel (COPV) made from continuous recycled carbon fiber tow
- new ways to recycle carbon fiber composites.

The experimental work is documented and could potentially be used as a basis for a new standard. Commercialization can occur rapidly when conditions are right. Many classes of smart materials had long incubation times, but saw rapid commercialization once the time was right. Other manufacturing costs need to be cut. Carbon fiber itself, at $28/kg for standard modulus fiber, represents just 22% of the cost of
a final CFRP part. Additional advances are needed to reduce capital, labor, energy, resin and processing costs, which together make up the remaining 78%.

Interest is increasing on hybrids and self-healing materials. With applications in composites and coatings, self-healing materials are set to be the next frontier for smart materials. Such materials automatically repair damage to themselves through one of several chemical mechanisms. CFRP developers will have to continue the pace of innovation to overcome the high cost that has so far limited the material to less price-sensitive markets like aerospace and sporting goods. Effort has been put globally on recycling and use of recycled CF.

Work Package 7: Manufacture of Proof of concept

The background to the purpose of the FIBRALSPEC FP7 project is explained in terms of addressing the research requirement to assist the opening of new ways for the industrial production in Europe of carbon fibres, as well as their functionalization for targeted applications, at an affordable cost. Bringing together a balanced consortium of research organisations, industrial companies and SMEs, the project combined technical skills within advanced materials science with practical expertise of product innovation. The consortium set out to develop innovative and streamlined processes for producing continuous PAN-based carbon fibres, testing the new fibres, manufacturing new laminates and applying them to specific demonstrators as proof of concept in the scientific research and innovation work being undertaken.

WP 7 “Manufacture of Proof of Concept” within FIBRALSPEC concentrated on the construction of two (2) demonstrator products; (i) high technology / small scale, in the form of supercapacitors, and (ii) medium technology / large scale, in the form of the Rapid Deployment Secure Emergency Shelter (RDSES). The RDSES is a shelter for use within in the humanitarian aid environment. It provides a more rigid, weatherproof shelter system with advantages over tents as an accommodation offer. The RDSES offering from the FIBRALSPEC project has a much longer projected life expectancy of up to 25 years (with maintenance), compared to the short life expectancy (12-18 months) of the common used canvas tent alternative.

The Methodology section of this report explains step-by-step the development work undertaken by WP7 partners GSG and AP&M in relation to (ii) above, the RDSES in carbon fibre (cf). Overall, this information forms FIBRALSPEC project’s deliverable report D7.2 which was developed by WP7 Task 7.2 lead GSG with input from Task partner AP&M working also on the practical development of the RDSES-cf demonstrator.

Following the FIBRALSPEC DoW, WP7 Task 7.2 partners completed the manufacture of the RDSES proof of concept unit in carbon fibre. After an initial mould for the RDSES was created, it was used to commence the process of refining the RDSES design, resulting in the final mould which was used to create the scaled carbon fibre proof of concept unit. In parallel with this practical activity, an alternative use for the RDSES unit was explored and developed, resulting in the full Business Plan for TheSleepZone Concept which incorporates the unit, as the SEA (Secure Event Accommodation) Unit, to the overall outdoor event service. Concluding, GSG will be able to use the new knowledge gained from the FIBRALSPEC project and incorporate it into the design and manufacture of future shelter systems. There is opportunity, by working with the humanitarian aid organisations, to implement the use of a new generation of lightweight, stronger, composite material shelter systems into disaster situations. This will also benefit the commercialisation potential for TSZC in due course. Development of composite materials reinforced by carbon fibers is very actual and perspective for the composites of multifunctional applications. Carbon
fibers are characterized by the complex of high physical, mechanical properties low specific weight, increased electrical and thermal conductivity, heat and chemical resistivity which ensure high properties of composites reinforced by these fibers and their using in aerospace, machine building, wind energy, electrical engineering, electronics and etc. Thermoset resins (epoxy, polyamide, phenol) and thermal resistant thermoplasts which allow to realize the properties of carbon fibers in composites are usually used as polymer binders for carbon plastics. The main part of structural carbon plastics is produced on the base of epoxy binders due to their high adhesion to carbon fibers and small shrinkage and cohesion strength of carbon plastics in the cured state. For better realization of the properties of both carbon fibers and polymer composites based on them and obtaining of the necessary level of service parameters of CFRPs, it is possible to use carbon nanostructured particles for modification of carbon fibers and/or polymer binders. The processes of modification of the surface of carbon fibers based on PAN precursors by carbon nanoparticles gave the possibility of increasing of strength and elastic modulus of carbon fibers and also enhancing of the properties of carbon plastic composites reinforced by such fibers.

Modification of the epoxy based binder by small amounts of carbon nanoparticles (about 1%-1.5%) can result to enhancing of adhesion interaction at the interface fiber-binder, increasing of the mechanical properties of carbon plastic and appearance of new functional properties of the carbon plastic in whole. So the main objective of the joint activity of IPMS and SDO Yuzhnoye within WP7 was to demonstrate the correctness of the main results obtained within WP2 and WP3 not only in laboratory conditions but also in industrial scale in particular on the example of pilot line on the production of special carbon plastic articles from nanomodified epoxy based commercial binder reinforced by nanoprofiled carbon fibers (nanoprofiling and nanomodification had been performed by carbon nanoparticles produced from the wastes of plant raw materials – pine chips).

This report presents the results of working places creation for an industrial line on production of composites from nanoprofiled carbon fibers, which makes it possible to improve the strength properties of carbon fiber by introducing nanoparticles, followed by the realization in composites. The mockups of carbon-fiber pipelines were manufactured by wet spiral winding of nanoprofiled carbon fiber, impregnated with nanomodified binder using technological equipment, specially designed in framework of FIBRALSPEC project. The main properties of mockups of standard structures are determined to confirm correctness of the project concept.

Work Package 8: Recycling

i. Recycling strategies for starting, intermediate and final waste materials generated in the FIBRALSPEC project have been presented. The philosophy of the recycling strategies is presented in the flow diagram in the attached file.

ii. The waste streams and the associated recycling strategies have been classified according to the manufacturing technique used to produce the preforms and filaments as illustrated in the attached file.

iii. A recommendation is made to specify a “quality criteria” for the fibres produced by the consortium.

iv. The selected production method for manufacturing composites produced and supplied by the partners to UoB is “Clean filament winding”. This manufacturing technique is at TRL has been evaluated previously.
on-site and was shown to be capable of reducing solvent consumption and the generation of mixed resin/hardener waste by 80%. Schematic illustrations of the conventional and clean filament winding techniques are illustrated in the attached file. The green credential of this new manufacturing technique will be positive for WPs 8 and 9.

v. The complexities in recycling composites are illustrated in the illustration in the attached file. The life cycle assessment and life cycle costs should be considered in WPs 8 and 9 (exclusively as part of the Fibralspec project).

vi. Preliminary work is reported on the influence of blending lignin with a high-performance resin thermosetting system. Scientific issues that need to be considered are detailed.

vii. The preliminary outcomes on melt-blending and wet-spinning are summarised.

• Solvent recycling reported by NTUA. Solvent waste was generated during the precursor purification and the fibre forming, and it was recycled via the evaporation for reuse.
• Lignin waste during the precursor purification has been reported by UoB in D8.1 Strategies for Recycling Starting, Intermediate and Final-Product Waste. The mechanical and physical properties of the composites consisting of epoxy matrix filled with lignin waste were determined.
• Recycling of the wastes of carbon nanoparticles generated in the production and storage and their applications as fillers for epoxy based polymers have been investigated by IPMS.
• Two methods of recycling carbon fibre composites were investigated by IPMS, UoB-CTM and APM-CSG.
• Grinding method. Recycling of carbon fibre composites by grinding was investigated by IPMS and APM-CSG. Composites were ground into carbon powders and the carbon powders were used as fillers for matrices.
• Pyrolysis method. UoB conducted the thermal analysis of carbon fibre composites by using thermogravimetric analysis (TGA). Recycled carbon fibres are still being investigated.

In the preparation of the fibre precursor, chemical solvent was used, and the solvent waste was recycled in NTUA using evaporation and distillation. The recycled solvent can then be re-used.
UoB continued on the investigation of the usage of lignin waste and carbon powders as epoxy fillers for manufacturing UD composites. The mechanical properties of the UD composites were measured by conducting tensile and flexural tests. Adding fillers into epoxy reduced the fibre volume fraction, because fillers located between groups of fibre. The mechanical properties, which were strongly determined by fibre volume fraction such as stress, tensile strength and modulus, were influenced. The strength and modulus of composites filled with recycled powders were lower than that of composites without recycled powders. Carbon powders were mixed with resin to make resin dough. The resin dough was then used to repair damaged fabrics. AP&M used the mixture of epoxy and carbon powders to make woven composite and single lap joint. The mechanical properties of woven composites and single lap joint were measured in UoB. Carbon powders have potential to be used as fillers for adhesive application.
IPMS investigated carbon powders used as epoxy fillers for the secondary composites. The tribology properties of the secondary composites were tests by conducting friction tests and their potential applications for bearing and water pumps were also investigated.
Work Package 9: Life Cycle Assessment

There is an increasing awareness of, and importance given to, environmental protection in many contexts. This includes the area of innovation and development of products and services, especially where new and advanced materials and processes are involved. The consideration of environmental impacts associated with this activity is of more interest now than it ever has been and there is a growing development of methods to better understand and address these impacts. GSG’s Rapid Deployment Secure Emergency Shelter (RDSES) is a shelter for use within in the humanitarian aid arena. It provides a more rigid, weatherproof shelter system with advantages over tents as an accommodation offer, and has a much longer lifespan, in comparison with the short life expectancy (12-18 months) of the common tents, up to 25 years. Within the FIBRALSPEC FP7 project, the RDSES features as a product being used for proof of concept in the scientific research and innovation work being undertaken. The focus of WP9 “Lifecycle assessment of carbon and hybrid RDSES” within FIBRALSPEC project is to quantify the green credentials of the reuse/recycling strategies via Life Cycle Assessment (LCA) and Life Cycle Cost (LCC) analyses. LCA for buildings is classified into three levels; 1. Materials and process selection, 2. Whole-building design, and 3. Whole-building assessment frameworks. WP9 Task 9.1 is focused on Level 1, using the commercial LCA software, GaBi. This report describes how WP9 partners undertook the various technical steps involved in LCA Level 1 for RDSES. The main constraints were assessed and basic material costs were calculated in order to predict shelter prices based upon material consumptions.

Within Task 9.1 activities, the following elements occurred:

-UoB undertook the LCA and LCC analysis to define the green credentials of the reuse and recycling programmes
-GSG assessed the lifecycle performance of the carbon and hybrid RDSES
-APM contributed to the composite assessment of the RDSES proof of concept.

The deliverable D9.1 reports the production of carbon and hybrid RDSES, which considerers;
- Shelter weight must be less than 500 kg
- The surface area of the designed shelter is about 30 m²

Five types of composites for RDSES, namely carbon composites, glass composites, flax composites, glass – flax composites and carbon – flax composites were investigated.
Based on the composite density, the shelter weight, the shelter wall thickness and the shelter price were predicted and calculated. For the shelter wall thickness of 6 mm 0.5 mm, the shelter weight was between 225 kg (flax and glass-flax shelter) and 275 kg (glass shelter).
LCA was conducted on all of the shelters. The carbon shelters gave higher environmental impacts than other shelters. Till now, in literature no direct correlation between construction material and shelter’s sustainability was found. However, the appropriate design and material selection can drive the sustainability of the shelters. Both global and local construction materials can be used to produce sustainable solutions for post disaster reconstruction projects, with local materials having higher potential for low environmental impacts and cost and global materials having better technical performances.
Life Cycle Assessment (LCA) and Life Cycle Cost (LCC) on carbon composites that were filled with the
recycled materials, such as lignin and carbon powders was conducted; Uni-directional (UD) carbon composites were filled with recycled lignin and carbon powders. The mechanical and physical properties of UD carbon composites with or without recycled materials were reported in D8.2.

In WP 7, UoB, AP&M and GSG have investigated the composites for rapid development secure emergency shelter RDSES. Flax, carbon and glass in weave forms were used as reinforced materials. Hybrids of carbon - flax and glass - flax were also investigated. Recycled materials such as lignin and carbon powders were added into epoxy and then used it to impregnate carbon and flax weaves as core materials of the composite shelter.

UoB has conducted LCA on the carbon fibre reclamation via pyrolysis.

UoB has performed preliminary LCA and LCC for the production of polyacrylonitrile PAN nano fibres using Mechano-Electro-Spinner MES.

Recycled materials such as lignin and carbon powders can reduce the environmental impacts and cost of carbon and carbon hybrid composites.

UoB conducted LCA on carbon fibre reclamation through pyrolysis. The environmental impacts resulted from the pyrolysis of CFRP waste were much lower than those from the carbon fibre production. UoB also conducted LCA on the production of PAN nano fibres manufactured by the electrospinning. Composites are increasingly considered as a preferred material choice in designing and developing complex, consumer products, because they are easily shaped, lightweight, and are often perceived to be highly recyclable materials. However, the recycling of heterogeneous Composites used in such durable items is rather challenging, and presents very different scenarios to how simple products, are recovered via curbside or container recycling initiatives. While the technology to recycle CF Composites exists, their feasibility to do so from high level consumer or industrial applications is bounded by technological and economical constraints. Obstacles include the lack of a market for recyclates, and the lack of cost efficient recovery infrastructures or processes. Furthermore, there is a knowledge gap between manufacturers, consumers, and end-of-life facility operators. For these reasons, end-of-life composites are more likely to end up down-cycled, or as shredder residue and then landfilled.

Considering carbon fibre composites, a successful recycling supply chain would mean cheaper inputs for manufacturers, helping them to break into new markets for applications where the material was previously too expensive and compete better on cost and quality. Failing to develop this will leave manufacturers almost entirely dependent on imports, increasing their exposure to global price volatility. It also closes off the chance to export expertise in both recovering and reusing of fibres. There are three simple overarching principles for developing novel materials in a way that will make them circular economy ready:

a) Think ahead and identify, during the development phase, what could enhance or prevent the circular use of a novel material at mass market scale to avoid problems in recovering value and enable a longer and more useful life to be designed in at the outset,

b) Collaborate along the supply chain to make the most of opportunities to maximise a material’s value. Systems can then emerge that no single company could achieve alone. For instance, co-operation between reprocessors and manufacturers would make sure that recovered materials fit with existing production processes.

c) Public sector support. Publicly funded research for the development and deployment of novel materials must always take their end of life recovery into account. And public policy should promote the highest value, least environmentally damaging recovery systems rather than low value options like landfill and incineration.
Next generation materials, such as carbon fibre, further complicate recycling due to their inherent complexity and being difficult to separate. If circular economy considerations are not built in from the start for CF composite products, it could set back progress on reutilizing and valuable resources will be lost to the economy. Support should be given to commercialise the technologies proven at laboratory scale and develop new recycling technologies to recover high quality fibres from discarded products. It is also a business model challenge to secure sufficient feedstock for commercial reprocessing infrastructure. For the automotive industry which is the expected major user of CF composites, collaboration with manufacturers and end of life vehicles handlers may be the best solution to this improve the recyclability and develop recyclable polymers for use in new composites. This requires development of new polymers or new additives. It is also a business model challenge to secure sufficient feedstock for commercial reprocessing infrastructure. The High Value Manufacturing Catapult and the Knowledge Transfer Network help to develop the markets for these fibres. The need for a business response to global resource constraints is already recognised. Those countries and companies that meet this challenge will enjoy a competitive advantage at home and abroad.

Effort has been put globally on recycling and use of recycled CF. Circular economy considerations should be built in from the start for CF composite products, if not this could set back progress on reutilizing and valuable resources will be lost to the economy. With landfilling no longer an option in Europe, there is a pressing need to develop feasible solutions.

Due to the expected increased quantities in the next few years of CF composites waste, there must be sensible statutory control of landfill and effort to decrease high production costs in the recycling of CFRP. The existing processes of solvolysis and pyrolysis seem to be the most promising methods. So far, however, no method has been implemented where no significant downcycling takes place. France will become in a few years a major player in the production of CF. One of the difficulties remains the lack of standardization of materials, which hampers the generalization of the use of composites, whether glass fibre or carbon.

There will be a huge demand for well-trained composites professionals; technicians, engineers, designers. The skills required in the coming years will be constantly changed due to the rapid evolution of manufacturing processes.

End-of-life, in the context of manufacturing and product lifecycle, can be considered as the final stage of a product’s existence. The particular concerns of end-of-life, depend on the product and whether the perspective is that of the manufacturer or the user. For the manufacturer, end-of-life, involves not only discontinuing production, but also continuing, to address the market needs that the product addresses, which might lead to the development of a new product. For the business using the product, end-of-life, includes disposing of the existing product responsibly, transitioning to a different product and ensuring that disruption will be minimal. In this deliverable, the end-of-life of precursor materials, carbon fibers, as well as composite materials (end products), are considered, taking into account both the manufacturer and the end-user concerns.

Special emphasis has been given to supercapacitors developed by partner THALES and the Rapid Deployment Secure Emergency Shelter (RDSES) developed by GSG, which have been analysed through their whole life cycle, from the early stage of production until their End-of-Life. The initial stages of the RDSES life cycle have been evaluated in already submitted deliverables (e.g. D9.1) and include the impacts of the materials selection and processes used for the construction of the shelters. In this deliverable, the effects of repairing a shelter that has reached its end-of-life are assessed, via GaBi software, which was used also in the other relevant deliverables by partner UoB. Regarding the
supercapacitors, the whole life cycle has been evaluated and different scenarios for their disposal have been considered, using SimaPro software, by partner NTUA.

Moreover, a comparison in terms of their environmental impact has been reported between CFs that are produced using different precursors, to show the advantages of using the alternative precursor lignin instead of PAN, which is the conventional one. The eco-friendly precursor lignin, plays a crucial role also in the end-of-life of the final products, since the life cycle “closes” with an environmental benefit, due to the recycling of paper wastes for the isolation and reuse of lignin. The goal here is not only to confirm whether the recycling of the aforementioned products is or not a promise for optimizing the environmental benefits, but also to confirm whether the novel precursors used for the production of the CFs can reduce the environmental impacts of CFs when compared to state-of-the-art solutions. The aforementioned comparison was achieved also by the use of SimaPro software, by partner NTUA.

The environmental consequences of FIBRALSPEC products were assessed. Specifically, a comparison between conventional precursors for CF production and alternative “green” resources was achieved, showing that the use of lignin has lower environmental impacts in general. However, when CFs are produced from lignin, the environmental impacts increase and become greater that the respective from PAN, since additional steps are required for the purification and functionalization of lignin in order to be processible for the CF manufacturing.

Moreover, D9.4 report presents the LCA and LCC on the end life of a composite shelter. Two scenarios were investigated. The first scenario is to repair an old composite shelter by doing composite repair. Glass of carbon woven composite was added into the old shelter. The thickness and the weight of the old shelter will increase due to the composite repair. Repair with carbon composite is more expensive than that with glass composites. The environmental impacts resulted from the composite repair are much less than those from the new composite shelter. The second scenario is to grind an old shelter into powder. The powder could be used as filler for producing new composite shelters. For fair comparison, a new shelter will be purchased in the second scenario. The second scenario is much more expensive than the first scenario. The environmental impacts resulted from the second scenario are also much higher than the first scenario. The high environmental impacts in the second scenario are generated from manufacturing a new shelter.

Finally, the LCA and end-of-life treatments of supercapacitors was conducted, taking into account two possible scenarios for supercapacitors disposal; the 100% landfilling and the recycling od supercapacitors packaging. It was proved, that the recycling procedure offers environmental benefits in the whole life cycle of this product.

Work Package 10: Dissemination, Exploitation and Standardization

a) Dissemination:

Dissemination strategy was a vital importance and promoted the main message of the project and represented the main actions and tasks to efficiently disseminate the project results. It helped to define how to achieve a critical mass of stakeholders and widen their horizon with the information of the upcoming breakthrough innovations, challenger and goals of the project. All project partners were actively involved in dissemination of the project results and were obliged to ensure the maximum high quality exposure to the project achievements.

The strategy focussed on both internal and external dissemination and the strategic and operational direction of the strategy to fulfil the aim of raising awareness of the project.

Within the internal dissemination, the following tools were presented:
b) Exploitation:
The EXPLOITATION PLAN was describing the strategy and the concrete actions for exploitation of the project’s results. Information was presented by exploitable result to facilitate usability. The industrial partners supported to ensure the impact of the research efforts, convincingly proving scalability towards industrial needs of two high demanding applications, namely medium technology – large scale (Rapid Deployment Secure Emergency Shelter (RDSES)) and high technology – small scale (supercapacitor), which will have significant impact on EU market providing fundamental know-how in terms of high ended value CF products. Also an exploitation workshop was carried during the last period of the project (M40) and all partners identified their Key Exploitable Results and the potential exploitation routes. Industrial take-up of the technology developed within FIBRALSPEC plays a central part in the exploitation of CF composites applications. To maximise the likelihood of success, the project provided customer-friendly access to the developed technology. At the same time the interests and investments of the partners were actively pursued by protecting any intellectual property developed within the project through secrecy agreements and by not disclosing any confidential information. The protection of technology applied both to device concepts and to developed process technology and became the shared property of the partners involved.

The importance of the EU to be self-sufficient in carbon fibres cannot be over emphasised. Whilst the aerospace and defence sectors can setup long-term purchasing contracts with suppliers (most of whom are outside the EU), SMEs cannot do the same and they are at the mercy of price fluctuations. The unique aspects of this project were: (i) The consortium acquired intimate knowledge about the precursor chemistry and the processing conditions that are used. This enabled the consortium to tailor the interface as required for specified matrices and end-use applications. It is generally appreciated that the nature of the interface dictated the general properties of composites. (ii) An intimate knowledge of the precursors and the analytical techniques that was used to characterise the conversion of the precursor at every stage of production enabled a fundamental understanding of the mechanisms associated with the chemical conversion processes from the precursor to the fibre. The consortium was also able to study the cause-and-effect associated with internal and surface flaws. (iii) The intimate knowledge of the starting materials enabled pragmatic and cost-effective strategies to be specified for the reuse and recycling strategies for each waste-stream, as opposed to concentrating of the recycling of the recovered fibres from the composite. Finally, (iv) the investigation into bio-based precursors set the standard for moving away from the reliance of petroleum-based precursors.

c) Technology transfer:
Technology transfer is the process of transferring skills, knowledge, technologies, methods of
manufacturing, samples of manufacturing and facilities among institutions to ensure that scientific and technological developments are accessible to a wider range of users who can then further develop and exploit the technology into new products, processes, applications, materials or services.

The final Objective was to move promising research topics into a level of maturity ready for bulk manufacturing or production.

Actions that took place leading to Technology Transfer were:

- Follow market trends in order to establish a beneficial commercial value for final products
- Technology watch of the project external environment
- Investigate and propose appropriate mechanisms for TT e.g.
  - Licensing agreements
  - Patents
  - Setting up joint ventures and partnerships
  - Start ups etc.
- and Keep an update on events for TT Log

Work Package 11: Management

In addition to the Periodic Reports, which were prepared for each single reporting period (M18, M36 and M48) and include both the technical and financial reporting for a particular reporting period, two Management Reports (M24, M48) were drafted and made available to the Commission, by uploading to SESAM at the EC Participant Portal, to follow up and evaluate the operational management of the planned work, including main measures and events taken, results achieved, technical problems, delays or deviations, unforeseen changes and the use of resources, in relation to the set time schedule and work plan as given in Annex 1 of the GA (DOW). These management reports provided a 24-monthly update of all relevant organizational, administrative, financial, and technical, information and communication aspects also regarding the interaction and cooperation with other projects, clusters and relevant stakeholders.

Potential Impact:

Economic impact

The FIBRALSPEC technologies have clear market potential and will have a strong impact on the economic prospects of the SME participants via two routes:

- The industrial partners will use the technology directly in their own manufacturing operations and/or directly in the services they provide.

- The industrial partners will market the technology through process licensing to other manufacturing organisations (via product type, market area, geographical region).

The FIBRALSPEC project well addresses all the impacts listed in the work programme of the Call (Table 1). The project aimed in increase of competitive power of European CF sector and especially that of the industrial partners. Industrial partners are more sensitive in the conditions of growing competitiveness because of the limited resources and access to modern RTD facilities. Benefits for the involved SMEs and downstream producers.
The existing concept “Rapid Deployment Secure Emergency Shelter- fibreglass” (RDSES-FG) designed and developed applying traditional composite manufacturing techniques has been extended through FIBRALSPEC by employing the use of carbon fibre, reducing the unit mass while at the same time increasing rigidity, durability and end user usability, resulting in Rapid Deployment Secure Emergency Shelter carbon fibre (RDSES-CF). The current RDSES-FG unit concept and all of its components weigh in at 500 kg. The target is to reduce the current design weight by 60% to approx. 200kg per RDSES-CF unit. A reduction of wall thickness by 50% from 60mm to 30mm would reduce mass per unit. This would allow greater numbers of stacked/nested units to be transported per shipment, therefore making significant gains in transport efficiency. An increase in components ability to resist damage would reduce the maintenance required during and after installation. Any increase in component rigidity would allow more efficient on-site installation. Less flexing would reduce the requirement for time consuming site preparation / ground levelling. As these RDSES-CF units will be sited in circumstances that may not have the use of lifting equipment, the target of 200kg reduces the amount of on-site manpower / manual handling required for unit assembly. Additionally, the use of carbon fibre would greatly assist in the end-of-life recycling. The shelter design could be made out of GRP sandwich; using strips of the new carbon fibre to stiffen the GRP panels will have significant impact for a carbon/glass combination where the structure is inflated from a packed state (deployable structure which can be parachuted to the disaster area and then inflated). Supercapacitors technology is being developed through this project and this fact creates huge economical potentials as well because this market looks promising with opportunities in transportation, electronics and energy industry. The knowledge gained from this project could create qualified personnel, decrease unemployment and contribute to EU’s economy. Another aim of this project is to enhance competitiveness and the exports of European industry by defining new international standards in CFs field. The increase of employment of high qualified personnel is also expected (Table 2).

Environmental impact
The replacement of less environmental friendly technologies will occur with more intelligent systems. Material waste losses will be reduced due to reliability and in service performance of components and reduced corrosion activity. Safer working conditions will also be ensured and carbon dioxide emissions will be limited due to savings in materials.

Social Impact
Within the area of Humanitarian aid the issue of protection of vulnerable people in disasters worldwide is critical. RDSES design and manufacturing process also could develop onsite manufacture right in the heart of the affected territories using containerized factories. This will allow a suitable programme of production using local labour, local resources were available and therefore stimulating local economic activity

List of Websites:
http://www.fibralspec.net/

Last update: 20 June 2018
Record number: 231600