New cost-effective and sustainable polyethylene based carbon fibres for volume market applications

Reporting

Project Information

NEWSPEC

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Final Report Summary - NEWSPEC (New cost-effective and sustainable polyethylene based carbon fibres for...
Executive Summary:
Carbon fibres (CFs) are the most efficient reinforcing materials in modern composite applications that require high specific stiffness and strength. In this context, the development of cheaper and more sustainable fibres is nowadays of paramount importance and this is further exacerbated by the current high demand for CFs.
NEWSPEC was aimed at the production of low cost CFs through very promising polyethylene (PE) precursor. PE, as precursor, has very much interesting technical features but it presents also several technological challenges that are not yet solved by state-of-art solutions. Therefore, NEWSPEC was proposing some crucial R&D breakthrough which are associated to high impacts as well as high risks. NEWSPEC has generated in total 74 accepted Deliverables with 15 achieved Milestones. The Project ends with 2 published international patents, one related to the innovative process for solid-phase sulfurization of PE precursor and one related to plasma surface treatment process, as well as a number of high level publications and conference presentations. Besides dissemination, NEWSPEC organized two very successful training courses for young students and engineers that provided a unique opportunity to put in contact the academic sector and the carbon composite industry, thus offering to young people both a high level formation and a qualifying experience. During NEWSPEC some innovative and working prototype machines for continuous endless CFs processing have been designed, developed and tested, in particular: a melt-spinning process equipment capable of semi-industrial manufacturing of PE fibres, the gas phase sulfurization equipment (called “SULFI”), a double-spot plasma equipment for CF surface functionalization and a remote Raman probe for in-line monitoring of mechanical features of the produced CFs.
The Project has generated fundamental know-how related to low-cost CFs manufacturing from innovative precursors, such as polyethylene. Before NEWSPEC, many unknowns were present in the chemistry of PE material and several risks related to its handling were not yet well defined. NEWSPEC made a big step forward in this direction: during the project a huge number of compounds were tested, many diverse solutions were tried and lot of attempts were done. Many of them failed, but all those failures helped us to find the right direction for a new PE-CFs manufacturing path.
Finally, NEWSPEC ends with a relevant number of exploitable results which are not only interesting for carbon fibre manufacturing industry but also to other stakeholders such as advanced polymer makers, textile producers, plastic compounds suppliers, machines manufacturers and various end-users of the carbon composite material chain.
In conclusion, NEWSPEC project has put good and strong basis on which to build up a new way of manufacturing low-cost PE carbon fibres. The perspective of this Consortium is to finalized the experimental work at pilot level scale in order to definitively prove the industrial feasibility of our idea. Once this will be done, it will be necessary to aggregate investments for a process scale-up to a pre-industrial manufacturing line with a limited production capacity (e.g. some tons per year), that will be necessary to fully evaluate the technical and economic viability of this technology.
However, the extensive use of CFs in large volume market applications is severely limited by the current market price of CFs - over 20 euro/kg - that results from the dependence on petroleum-based precursors and the laborious, expensive and often non-environmental friendly conversion processes.

In this context, the development of cheaper and more sustainable fibres is indeed nowadays of paramount importance and this is further exacerbated by the current high demand for CFs.

NEWSPEC was aimed at the production of CFs through very promising precursors, such as polyethylene (PE). PE presents interesting technical features like high carbon yield (around 70%), high processability, flexibility and very competitive cost (~2 euro/kg) with respect to PAN precursor that may result into a precursor cost saving of up to 70%.

The target technical features of the PE-CFs should be: 250 GPa tensile modulus, 2 GPa tensile strength, 1% elongation, 10 microns diameter, and 1.8 g/cm³ density. The resulting CFs must be also homogenous with very smooth fibre surfaces (low surface roughness) and residual sulphur content as low as 1%wt (or even less upon optimization of carbonization time and temperature).

Considering the overall CF production cycle, the estimated final target cost of PE-CFs was expected to be around 10 euro/kg, which means a cost reduction of 30% with respect to comparable PAN based fibres.

To prove overall environmental and economic sustainability, specific LCA and LCC have been implemented within all phases of the project.

Main breakthroughs proposed by the NEWSPEC project were:

1. Optimized LDPE and HDPE fine fibres processed by melt-spinning (instead of conventional wet spinning) which has been never attempted so far. Besides, bio-PE precursor was used. Bio-PE derives from dehydration of bio-ethanol to ethylene and then polymerization to PE and this precursor offers significant advantages:
   a. Bio-ethanol is largely available today - its production has rated 100 bln litres in 2011 (EOCD-FAO) – being mostly produced as a fermentation by-product of sugarcane and other annual food crops (corn, wheat, barely).
   b. Bio-PE exhibits the same chemical and physical properties as PE from fossil resources and good purity;
   c. Its energy consumption in the cycle (biomass to polymerization) is reduced by about 70% when compared to the petrochemical cycle;
   d. Is a sustainable, renewable and ecological precursor: producing 1kg of bio-PE captures 2.5 kg of CO2 from the atmosphere.

A major challenge was not only to spin fine continuous fibres (up to 12k tows) but also to make them compatible with subsequent stabilization and carbonization steps.

2. For PE stabilisation an original sulphur vapour phase stabilization method assisted by Electron Beam Curing (EBC) that introduces heteroatoms at the precursor stage has been proposed. The sulfuration leads to thiopolymers, which can condense to polyaromatic systems very similar to PAN. The new approach has technical, economic and ecologic advantages thus ensuring very innovative and flexible development of new CFs.

However, this required the development of a radically new equipment for continuous fibres treatment which represents the main technical challenge of NEWSPEC project.

A main attribute of this project is the use of an available High Performance Fibre Centre (HPFC) pilot scale...
facility that allows design and optimization of endless CF processing and easy scale up to a larger size industrial plant.

3. Novel strategies for the reduction of the graphitisation temperature via the use of nucleation agents such as cellulose nanowhiskers (CNWs), CNTs, graphite powders or Graphene Oxide (GO) have been envisaged. Lowering the graphitization temperature from 1500° to 1200°C can contribute to cost reduction of about 15-20% with respect to typical PAN process. We were also expecting that the NPs provide increased mechanical properties of fibres thus contributing to facilitate the PE sulphorization step.

4. The partners explored the possibility of CFs surface modification. Surface functionalisation is a necessary ingredient for the efficient use of CFs since the strength of the interface governs the properties of the resulting composite materials. Within NEWSPEC, several innovative functionalization routes for the surface treatment of PE-based CF have been explored: (a) atmospheric plasma technology for controlled oxidation and grafting of other selected functional groups to the surface; (b) new methods of rapid room-temperature grafting on graphitic surfaces using specific surface-attacking chemicals. These new approaches were confronted with conventional electrochemical treatment. Successful replacement of conventional methods by either (a) or (b) will further reduce the overall cost of the fibre and make the whole fibre process more sustainable.

The structure of the new CFs had to be characterised with various spectroscopic methods and structure-to-property models have been developed. The efficiency of the various treatments shall be also assessed with specific techniques. Surface techniques like XPS are employed to identify the implanted chemical species. Furthermore, pertinent analytical treatments based on shear-lag analysis (for polymer composites) are employed to assess the transfer length for efficient reinforcement and the interfacial shear strength of these systems.

5. One of the NEWSPEC objectives was to develop and optimize one transportable confocal micro Raman system which has to be used on the processing line for monitoring the various steps of CF synthesis. An automated system was developed and installed in the HPCF pilot line and it is capable for acquiring spectra from different locations of the production line. The proposed system offers significant capabilities for a non-destructive quality assessment in the fibre production line. Parameters such as the degree of crystallinity, carbonization and graphitization of CFs, the level of disorder and structural information will be provided in-situ.

Carbon composite coupons and prototypes have been manufactured and tested by the end-users during the lifetime of the project to ensure the validation of CF functionality for the final components with this ensuring proper exploitation of results.

NEWSPEC has involved specialized end-users as manufacturer of carbon composites, both CF reinforced and carbon ceramic matrix composites, in each of above mentioned segments: aerospace, automotive, wind, oil/gas transportation and storage. Those sectors were identified as some of the most promising either in terms of PE-CFs technical requirements and in order to maximize the impact of the project results.

Project Results:

OVERVIEW

The NEWSPEC programme has run for 54 months (48 months of the original workplan plus a 6 months prorogation) and the activities were split down in the following Work Packages:
As outputs, NEWSPEC has generated in total 74 accepted Deliverables with 15 achieved Milestones. Many of the original objectives have been successfully met, all except that the foreseen validation and functional demonstration of PE-CFs in real industrial environment. This work has been not possible due to the fact that the Project produced only a few hundred grams of PE-CFs which were not optimized for making big size industrial samples, thus the corresponding WP7 has been cancelled from the work programme.

Nevertheless, NEWSPEC ended with 2 published patents and a number of high level publications and conference presentations. Moreover, in the framework of NEWSPEC some innovative prototype equipment that will be described in detail in the following sections have been designed, developed and tested, namely:

i) melt-spinning equipment for semi-industrial manufacturing of PE fibres at DITF,
ii) the gas phase sulfurization equipment by DIENES (called “Sulfi”),
iii) the remote Raman probe by FORTH, and
iv) the double-spot plasma equipment by VITO.

The development, validation and assessment of the PE-CFs has been thought according to three phases that correspond, to related technical WPs:

1. Lab phase i.e. establishment of PE-CFs processing at batch level (WP2);
2. Pilot phase i.e. upscaling of endless carbon fibre manufacturing at HPFC (WP3);
3. Demo phase i.e. manufacturing of CF reinforced composites and material coupons (WP6);

The NEWSPEC programme started at relatively low TRL. Thus, a remarkable amount of work and effort was necessary to investigate the PE-CFs manufacturing on batch (stationary) process (WP2). This work included several issues such as: a) precursor preparation, b) optimization of the parameters for melt spinning, c) precursor stabilization (cross-linking) and d) carbonization (best temperature profiles and treatment times in stabilization for high carbon yields and good mechanical properties of carbon fibres).

The work done in WP2 and its deliverables have paved the way for upscaling the CFs manufacturing to a continuous process (endless fibres) having well defined all required process steps, methods and parameters. After completion of batch processing phase of PE-CFs, the NEWSPEC programme run into endless CFs manufacturing at HPFC pilot facility in Denkendorf (DE).

For the manufacturing of endless PE-CF it has been necessary to complete the existing HPFC pilot line with specific equipment for PE precursor handling and stabilization. In fact, NEWSPEC aims to replace the wet (acidic) oxidation employed so far for PE stabilization with a dry method that introduces heteroatoms at the precursor stage and a combination thereof with Electron Beam Curing (EBC) for PE cross-linking. This process was radically innovative and never attempted so far to stabilize endless PE fibres on a pilot scale level.

During NEWSPEC, DIENES was in charge to design and install the equipment for the sulphur gas-phase stabilization of the cross-linked PE-precursor. This has been a challenging activity, since there was no previous research experience and many technical unknowns were behind the corner. Nevertheless, thanks to restless commitment of DIENED and DITF, the equipment, called “SULFI”, has been installed at HPFC in April 2016 and begun to be operational in September 2016. This equipment has to fulfil the Machinery Directive 2006/42/EC EN 60204-1: 2007 (Safety of machinery - Electrical equipment of machines) and an independent technical audit was performed by TUV in August 2016 to ensure that the facility could operate without any safety risk.

WP6 aimed at the validation of newly developed fibres for composite manufacturing in selected industrial segments. Some composites coupons were designed, produced and characterized by ASTM standard
tests. However, due to the delay with operation of the pilot stabilization equipment and little availability of PE-CFs materials for the end-users, at the end it was possible to realize only very small samples size. In parallel, the end-users carried on activities of preparation of reference materials either using PAN type or other similar CFs. This can be used as a first test to evaluate and compare in the future the performance of the products made with the new PE fibres with those made with commercial one. Moreover, these tests can be useful to set up the prototype production process whenever PE-CFs will be available.

Aside to these activities two crucial WPs were related to the development of alternative surface treatments (like plasma and RT epoxidation) and to non-destructive structural and mechanical characterisation of fibres by implementing real-time in-line tools (remote Raman probe). Both WPs were completed with success.

In the following sections we will present the main technological achievements of NEWSPEC. For obvious reasons related to the exploitation of the IP, some fine details are kept undisclosed.

**BIO-PE PRECURSOR DEVELOPMENT**

The preparation of CF from alternative precursors based on the known systems as lignins, cellulosics, and polyolefins were under investigation since decades. Polyethylene has the advantage of continuous carbon chains in its polymeric structure, which may transfer in good carbon fiber properties. Besides, it has advantages in terms of processability and low cost.

A main achievement of NEWSPEC has been the formulation of best bio-PE polymer precursors for carbon fibre processing. This activity was mainly carried out by TECNARO and DITF in collaboration with UNEXE.

During the Project, several PE materials from different sources (bio-based, oil-based and recycled) have been investigated. After preliminary analysis both recycled PE and oil-based PE were discarded due to uncontrollable quality of the polymer, presence of impurities and unmatched processing features. So, the partners focused on bio-based PE compounds that were developed by TECNARO under the own brand name of ArboBlend (AB).

The melt-spun trials of different PE compounds result in stable fibers melt-spinning with low orientation of the polymer chains. An additional stretch of the fibers results in a strength of up to 50 cN/tex and orientations up to 95%. Spinnability of the HDPE types is better than in the case of LLDPE, but also high molecular weight could be processed. The results achieved in the lab phase allowed verifying the following hypotheses:

1. LLDPE gets something higher carbon yields (up to 47% for carbonization step from sulfur-rich precursor) but less carbon fiber properties;
2. The HDPE got better carbon fiber strength with maximum of 200+/-40 GPa Modulus and around 1 GPa of tensile strength;
3. LLDPE has some drawbacks in melt-spinning (no low-viscosity type available, overall less stable spinning);

for those reasons HDPE was selected for the upscale in the pilot phase. Thus, as result from work in WP2 both polymers AB1639 X and AB1385 X were melt spun at the pilot plant at HPFC.

The compounding and Meltspinning of PE precusor was realized in a semi-industrial scale with up to 8 kg/h raw precursor fiber production volume (Figure 1). The process was done with 250 filaments at the time. The higher filament numbers necessary for typical carbon fiber precursors from 1,000 up to 12,000 tows were achieved by joining the yarns together (Figure 2 and Figure 3). The melt-spinning process in pilot-scale is stable and could be run over some hours without problems with production volumes of...
theoretical 70 tons/year on the pilot line. Lot of efforts were done by DITF and TECNARO to set up and optimize the working conditions in order to achieve the best quality PE fibres for CFs making. During the entire NEWSPEC programme an impressive number of 350 spinning trials have been performed. This means, on average, a spinning trial every 4.5 days (i.e. one trial per week). In particular, from November 2015 to October 2017, the melt-spinner has worked almost continuously to produce PE fibres for NEWSPEC. We calculated that about 710 kg of PE fibres were spun during the pilot phase.

Most of this material has been used for subsequent stabilization and carbonization trials on the pilot line. At that level we observed that some crucial improvements of the PE matrix were necessary. In fact, due to the missing thermal stability of the PE precursor in the sulfur stabilization chamber above 240 °C it was necessary to modify the PE matrix. Therefore, two paths were investigated: i) the addition of nano-agents to improve the fibre crystallinity (see next section), and ii) the melt-spinning of bicomponent fibers. The best results were achieved by using the second strategy, in fact the resulting fibers were mechanical stable up to 270 °C. However, the bicomponent spinning was limited to the existing spinning line at DITF to only 30-40 filaments at a time.

PE PRECURSOR MODIFICATION BY NANO-AGENTS AND METHODS FOR VERIFICATION OF DISPERSION OF NPS INTO A THERMOPLASTIC MATRIX

One of the goal of NEWSPEC regarded the precursor modification by nano-agents. Innovative strategies for the reduction of the graphitisation temperature via the use of small amounts of nucleation agents such as carbon nanotubes (CNTs), CNWs and fine graphite powders have been explored. This research area was almost unexplored and so have high risks of success, especially because of the challenging dispersion of nano-agents into the polymer matrix. The main goal was to perform a selection of suitable agents (done at the beginning of the project) and perform laboratory scale mixing trials. This work was started with transforming of natural graphite powder (GP) into graphene oxide (GO) using Hummers' method, several dispersion trials were performed but this method was abandoned because leading to poor results. Cellulose Nanocrystals (CNCs) have been successfully dispersed into the PE matrix. For this, UNEXE has developed, on lab scale, a method for mixing CNCs into the PE matrix upon freeze drying and grinding of PEO:CNC modified compound (Figure 5). This approach has been tested in cooperation with TECNARO and DITF. PE master batches with already dispersed Carbon Nanotubes (MWNTs) were acquired from suppliers upon specifications.

Another idea of NEWSPEC partners was to use nano-agents to improve the crystallinity of the PE. The best four samples of UNEXE prepared precursor fibers have been tested in the continuous stabilization process. There were 2 samples with CNT’s included and 2 CNC’s. Samples with CNC material included could be processed also with lower filament numbers as 250 single filaments in the yarn (Figure 6, top). The fiber gluing was reduced in those samples but not removed fully. Cross-cut and polished samples showed the not molten, but still sintered filament surfaces of the samples after heat treatment (Figure 6, bottom).

A main challenge was to verify the dispersion of the incorporated of CNC and CNT in melt compounded thermoplastics. UNEXE has then developed a now quantitative method based on confocal Raman mapping combined with chemical images and image analysis to provide a spatial distribution of CNCs in high-density poly-ethylene (HDPE) composites. The dispersion of nano-agents in the PE matrix was verified by converting a Raman spectrum into a “chemical map”.

The combination of Raman imaging with image analysis provides rich information regarding the mixing of
HDPE with CNCs, and also the agglomeration of the latter in a compounded composite. CNCs are found to form different agglomerated islands within the HDPE dependent on the drying process used for their production. Raman images provide wider information about the composite quality supported by a detailed chemical quantification and a basic morphological feature of the material. Data obtained from the Raman images are used to quantify the degree of mixing between CNCs and HDPE and the degree of aggregation of CNCs. It is worthwhile noting that the average ratio of agglomerated CNCs to CNCs within an area where mixing with HDPE has occurred can be used as an indicator of aggregation.

Figure 7 illustrates the protocol for the distribution/mixing verification containing three stages:
- acquisition of the Raman spectra - using confocal Raman (WITec Alpha300 R)
- data processing and generation of chemical images - using WITec Plus software
- image processing - using Image J software

The method has been published by UNEXE in a series of papers and conference contributions (see e.g. A.E. Lewandowska and S.J. Eichhorn, J. Raman Spectrosc. 2016, 47, 1337–1342).

FROM PE PRECURSOR TO CARBON FIBRES

1) Innovative process for PE precursor stabilization

PE as carbon fibre precursor is not new, the first patent by Sumitomo Chemical Co Ltd dates back to 1974 (US05438704) and was proposing a process for sulfonating PE fibres with chlorosulfonic acid, sulfuric acid, fuming sulfuric acid or a mixture of two or more kinds thereof. This path has been further explored by ORNL (UT-Battelle LLC, US13628463 2012-09-27) who set up a pilot facility in Oak Ridge, Tennessee in collaboration with DoW Chemical. However, in spite of their attempts, the wet treatment seems to be impracticable from an industrial point of view.

The main challenge of NEWSPEC was to replace the wet (acidic) oxidation employed so far for PE stabilization with a dry method that introduces heteroatoms at the precursor stage and a combination thereof with Electron Beam Curing (EBC) for PE cross-linking (Figure 8). The new process has been patented by DITF in 2016 (WO2016170081 A1).

Prior to perform trials at the pilot line, single fibre filaments were stabilized using a small home-made laboratory equipment and the best stabilization parameters for preparing the PE carbon fibres were identified. This work run for over 24 months during the lab phase. At the end of that phase, we demonstrated that the process could work (proof-of-concept principle, TRL 3 equivalent). Moreover, the best stabilization method for preparing PE fibers avoiding residues on the precursor fiber surface was found to be a treatment with sulphur-saturated nitrogen atmosphere at a temperature in range of 240-280 °C. An increasing EBC treatment dosage improves in some way the stabilization quality, so no crystalline by-products could be detected in carbonized samples, when the dosage is above 400 kGy. The stabilized precursor has good mechanical stability comparable to PAN-based precursor fibres. The well-defined density of 1.77 g/cm³ and the high temperature-resistance to at least 500 °C in nitrogen not independent from the PE source is a sign for a well-defined polymer structure in the precursor fibers.

We must point out that such a new process requires two crucial ingredients, which were all in all the main focus of the entire programme:

i) First, the process reaction works only at high temperatures (260 °C); yet, PE has a relatively low melting point (130-140 °C). EBC induced crosslinking can partially alleviate the problem but to prevent melting of the PE the precursor needs some additional modification to avoid the sintering of endless fiber filaments
during the stabilization. Thus, the process only works together a specifically designed precursor which makes the NEWSPEC foreground pretty much unique and very strong.

ii) Second, in order to process endless CF fibres, the process requires a special equipment for the sulphur gas-phase stabilization. This is perhaps the most relevant technology created during the NEWSPEC programme, as described in what follows.

2) The “SULFI” equipment

The treatment of a PE yarn in enclosed inert (nitrogen) atmosphere saturated with sulfur at uniform high temperature - up to 270 °C - for a long time (several minutes to hours) requires, as said, a special apparatus that has to be conceived, designed, constructed and tested. Additionally, gas exchange is necessary for maintaining an equilibrium condition in the gas atmosphere and all surfaces in contact with gas (including the godets) have to have at elevated temperature to avoid sulphur condensation. Moreover, the system has to be absolutely sealed to avoid H2S gases to refuge from the chambers and a safety SPS has to be implemented to make the system secure and to protect from wrong handling of the system. To do so, the system has to be equipped with reliable H2S sensors and fully automated. Starting from the very beginning of NEWSPEC, DIENES Apparatebau GmbH has been fully engaged in the development of this system, called “SULFI”, which consists of two chambers with i) heated walls and with integrated gas circulation with heating for uniform temperature and for reduced heat-up time; ii) yarn transport by heated and tilted godets in duo arrangement for simple string-up and to realize maximum residence time in compact space; iii) adjustable tilting angle to reach optimum degree of space utilization and maximum residence time also for different total yarn titer; iv) heating of godets for uniform treatment temperature of the yarn and for fast ramp-up to operation temperature of the installation; and v) handle exhaust gases.

During NEWSPEC, all the hardware, software and safety systems have been developed, including an external H2S exhaust treatment facility consisting in 4 tons of iron-catalyst who oxidizes H2S to sulphur (Figure 9).

This system was installed by DIENES in beginning of April 2016 at the HPCF facility and started to be operational from September 2016. Since then, DITF has started the system test phase which is still going on.

The development of SULFI was extremely challenging for many reasons:

a. The most important hurdle was to design a oven able to handle the H2S gas generated in the process which is highly toxic. This forced us to ensure that the sulphurization oven has to be absolutely sealed to protect people managing the process. Finding the right sealing was not easy.

b. Beside the toxicity of H2S is this gas very corrosive. Nearly all metal but stainless steel are not resistant against H2S. The high temperature of maximum 300 °C is also a technical challenge. Most of the sensors, which are available in the market, can’t cope with this requirement.

c. Another technical hurdle, which was solved, was that oxygen from the atmosphere which can get inside the chamber by leakage, cracks or the openings where the fiber is been transported into the chamber and out again. The influence of the oxygen reduces quality of the fiber. A second effect caused by oxygen is another dangerous problem. H2S gas with a temperature more than 260 °C will start burning without a spark when there is only a trace of oxygen inside the treatment chamber.

The SULFI equipment as whole had to fulfil the Machinery Directive 2006/42 / EC EN 60204-1: 2007 (Safety of machinery - Electrical equipment of machines) and to ensure full safety of the operators an independent technical audit has been performed by TUV who released its positive assessment report on
30/08/2016. So, the equipment operates today without any safety risk.

3) PE carbon fibres

Laboratory PE-CFs samples, consisting in short single filament CFs, were obtained in NEWSPEC at the end of the lab phase (month 24). This was not only a proof-of-concept of the new stabilization process, but also a demonstration that it could be possible to make CFs out of our bio-PE precursor.

HDPE compounds showed not significantly lower rest masses in carbonization, LLDPE have highest rest mass is 47%, which is near the theoretical carbonization rest mass of 54%. In summary, we proved that 73% carbon yield is possible now (against 85% theoretical carbon yield). By contrast, a classical PAN-based carbon fibre has around 45% carbon yield.

The resulting PE-CFs were non-brittle and could be processed without problems as single filaments. The carbon phase was well oriented. WAXS data showed a well-developed aromatic structure even in the precursor as in the resulting CFs. Best values achieved from HDPE AB 1387 X at a carbonization temperature of 2000°C with an average strength of 0.83 GPa, a modulus of 96.8 GPa and 0.9% elongation. Considering that the small samples were stabilized in lab conditions, these parameters were in line with expected targets on endless PE-CFs. Due to the long carbonization time and with applying tension this values should increase. Due to this result, better crystallinity maybe improves fibre quality. After having reached the WP2 milestone, which demonstrated the conversion of PE single filaments to carbon fibers, and having installed all the required equipment for endless fibre treatment (EBC, sulfi, waste gas plant, carbonization lines, etc.) the NEWSPEC work has continued at pilot line level.

However, continuous treatment of endless PE precursor fibres showed soon strong problems with the weakness of the crosslinked PE fiber bundles and a much stronger problem with the PE fibre sintering behavior (see Figure 10), due to the very high temperatures (above 200°C) required to activate the stabilization reaction path. Additionally the fiber sintering prevents the continuous stabilization and carbonization of the fiber bundles, because the sulfur can’t diffuse in the inner zones of the fiber bundles and the stiffening yarn tends to break a latest in a second stabilization step necessary to reach the full stabilization of the material, so the fiber samples became very stiff and brittle.

Besides this, several technical issues raised up during the SULFI tests, such as i) lack of sealing and leakages, ii) reliability and durability of temperature sensors, iii) blocking of coolers for sulfur condensation, iv) water inleakage into the chambers and oxygen inbrake into furnaces.

To solve those problems, NEWSPEC partners have been committed for 2,5 years trying to explore all possible technical solutions. Although there are still some minor remaining issues, nevertheless major problems have now been fixed and the pilot line is operational.

Low mechanical performance of the PE precursor was strongly improved by optimizing the crosslinking of the PE by heating the fiber direct after EBC radiation treatment. To solve the sintering of the crosslinked PE at temperatures above 200°C a lot of different but unsuccessful methods were tested including wet chemistry (waxes, vulcanizers, radical starters, and so on), plasma surface activation or thin coating of the PE fiber.

Finally, the solution was to radically modify the PE precursor (Figure 11 and 12). The new fibers showed no glued filaments in the standard hot-set test. Another positive aspect is that the stretched fiber only slightly shrinked. The main reason for this property of the stretched fiber is the higher orientation of the polymer matrix and its increase in tenacity. After the carbonization up to 1400°C the fibers stabilized at 240°C the REM images showed a porous cross section and a rough surface. A similar result on the surface is shown at the higher stabilization temperature, but the cross section of the fibers stabilized at
260 °C is much more compact after carbonization. The carbon yield was determined by TGA under nitrogen from 45 to 1400 °C with a heating rate of 10K/min. At 240 °C the stabilization was not fully achieved, so the carbon fiber was deformed and becomes porous as typical for such low carbon yields. At the time of the writing this report this type of precursor fiber were prepared in kg-amounts with 1,000 filaments and is now under testing for endless fiber stabilization and carbonization.

ALTERNATIVE PROCESSING TECHNOLOGIES FOR CF SURFACE TREATMENT AND FUNCTIONALIZATION

A relevant objective for the period is to design and develop new alternative processing technologies for CF surface treatment and functionalization (WP4). Surface treatment is the final step in the CF manufacturing process and it is crucial to tune the surface treatment to the application. In fact, there are several surface aspects that can be modified to maximise the interaction between fibre and matrix material (i.e. increasing the surface energy of the fibre, increasing the specific functional groups on the surface, increasing the surface roughness for mechanical interlocking).

During the NEWSPEC project two main technologies were investigated:

a) atmospheric plasma, that enables continuous endless CFs treatment; and
b) a novel method of epoxidation at RT only implemented at lab level on batch CF samples.

Those surface treatment technologies were developed, engineered and optimized by using PAN based CFs as model materials. They could be easily transferred to PE-CFs.

i. Atmospheric plasma equipment for endless CF surface functionalization

Within NEWSPEC, atmospheric plasma technology is proposed as an innovative technique for surface modification of CF (Fig. 13). Important advantages of this technology are the environmental friendliness (gas phase process), the flexibility in surface functionalisation (all types of chemical groups) and the possibility for in-line treatment.

Double-spot pilot prototype for plasma treatment and surface functionalization of endless continuous fibres (either fibre precursor or CFs) has been designed and built to allow the new surface treatment to be done on an industrially relevant scale. Dedicated configuration for unsized CF was fully developed by VITO who owns an international patent on it (EP3163983 A1, US20170125221, Pub. Date: 3/5/2017, Prior. Date: 28/10/2015, Inventors: Annick Vanhulsel, Hoof Erwin Van, Jan Cools).

The invention is related to apparatuses and methods for indirect atmospheric pressure plasma processing, in particular where the substrate to be plasma processed is kept remote from the plasma discharge zone. With indirect or remote plasma treatment of substrates, as opposed to in-situ plasma treatment, the substrate is not passed through the plasma discharge zone, where an atmospheric pressure plasma is maintained between electrodes and activated species are formed. Instead, the substrate is positioned at a location remote from the plasma discharge zone and the plasma-activated species are transported to the remote location where they are made to react with the substrate.

Remote plasma treatment is often preferred over in-situ treatment, in particular for cases in which in-situ plasma treatment would cause charging of the substrate surface and therefore undesirable interaction with the electric field of the plasma discharge. This is particularly the case for substrate materials having at least some degree of electrical conductivity, such as for instance carbon fibres.

The continuous plasma functionalization of endless CF in the pilot-scale system at VITO has been validated at pilot scale (TRL 6 equivalent) on different types of CF. Both sized and unsized CF of different tow size (5K, 12K, 24K) have been tested to assess the versatility of the continuous process. It was
demonstrated that within the parameters window, the atmospheric plasma process does not damage the CF which is an important prerequisite for potential use of the new technology in the CF production process.

The impact of the plasma treatment of CFs on the performance of thermoset composite materials have been evaluated in a collaborative action between VITO, Yuzhnoye YSDO and FORTH. Different types of commercial PAN-CF were plasma treated and a significant improvement in adhesion strength was observed in bending and shear tests. Based on these positive results, a larger volume of plasma treated CFs was produced for production of the YSDO demonstrators.

The equipment should be adapted and scaled-up to fit industrial facilities requirements. The fields of application and interested stakeholders are many. Plasma treatment today is used to clean, etch and functionalize plastic and metal surfaces in order to improve adhesion or prepare tailor made surfaces. Therefore, the double-plasma spot will be of interest for treatment and surface functionalization of various kind of fibres, especially for conductive fibres.

ii. Method for Room Temperature oxidation of CFs

Epoxidation is a chemical process involving reaction at room temperature (RT). It is utilised by the immersion of CF tows into an organic solution/bath of peroxide derivatives, such as peracetic acid or 3-chloroperoxybenzoic acid, for short times. Decoration of CF surface with oxygen moieties - and especially epoxy rings - at optimized grafting densities will yield to enhanced adhesion with epoxy-based matrices, through crosslinking reactions.

A specific methodology has been developed and tested by FORTH during the NEWSPEC programme. This process has been developed on a small lab scale, but it is promising to be upscaled at pilot level (Fig. 14).

The method has been evaluated by using commercially available untreated and unsized carbon fibres (TRL 3-4 equivalent). The results will also have to be confirmed by using PE-based carbon fibres. The nature of the epoxidation reaction, which is a pure interaction of carbon atoms with oxygen, cannot justify any correlation with the fibre precursor or the fibre production method.

The main conclusions based on the lab-scale experiments can be summarized as follows:

- The chemical reaction of epoxidizing the outer surface of the carbon fibres is successful. The process can be applied to carbon fibre materials;
- The epoxidation efficiency (as measured by the surface oxygen content) can be tuned and depends on both immersion duration and reagent concentration;
- The whole procedure (solution preparation and immersion) does not require any form of heating and can be utilized in room temperature;
- The epoxidation effect is present for immersion times well below than 5min;
- Preliminary results have shown an interface (between epoxidized CF and epoxy matrix) enhancement, compared to non-epoxidized fibres;
- After the epoxidation process the reagent (mCPBA) cannot be recovered. On the other hand, most of the solvent quantity (CH2Cl2) can be recovered and reused by evaporation.

When it comes to up-scaling the above process, there is a need to use a larger immersing sink containing the solution bath, a second sequential sink to rinse the remains of the epoxidation and a low temperature furnace (70-800C) in order to evaporate the liquid from the CF surface (optional, since CH2Cl2 can be evaporated in room temperature within a short time period).

Further R&D is necessary to optimize the formulation and verify the scalability. Pilot scale development is
possible upon such a validation phase and provided that the process would be economically viable.

FAST IN-LINE NON-DESTRUCTIVE TECHNOLOGIES FOR CF ANALYSIS PROTOCOLS FOR ANALYZING FIBER/MATRIX INTERFACE (REMOTE RAMAN PROBE)

NEWSPEC also aimed at the design, implementation and installation of a transportable remote Raman system for on line monitoring of the CF production process (Figure 15). FORTH was responsible of this activity.

The key features of this new equipment are:
- Simultaneous measurements in two different production stages
- Autonomous function of the monitoring system (electrical power only needed)
- Transportability of the instrument station
- Near-line installation
- Flexibility in adapting the system in different production line layouts
- Fully operated from remote location
- Remote image capturing and focusing of the beam
- Tailor-made chip for CCD splitting
- Easy focusing, easy image capturing by video camera
- No aligning required

The technology prototype (Figure 16) has been developed by FORTH in collaboration with Renishaw, a leading European industry in the Raman sector.

The Raman probe has been installed at the HPFC in April 2016. Then, all the protocols for analyzing fiber/matrix interface mechanical behavior with a fast, local, non-destructive method have been set-up and the system has been validated at pilot plant level on PAN-CFs for almost two years.

This technology today is fully ready to be commercialized and put into service. The Raman probe will be extremely interesting for Raman machine makers (Renishaw – UK) and various end-users e.g.: advanced polymers manufacturers, carbon fiber industry, composite industry and also research entities.

CARBON FIBRE REINFORCED COMPOSITES MANUFACTURING

1) Manufacturing and testing of CFRP coupons for composite winglet design and characterization of CFRP materials according to automotive standards

The main purpose of this effort was to prepare all necessary information for the design and manufacturing of a composite airplane winglet using the carbon fibers produced within the project. Two composite plates of the same size and thickness were supposed to be manufactured in order to produce the necessary coupons for the testing. The first plate was produced with the standard aeronautical grade materials while the second plate would use the same resin material and fibers produced from NEWSPEC project.

During the project, INASCO prepared a metallic mould for the manufacturing of plates for the samples and prepared the whole manufacturing (RTM), inspection (C-Scan) and testing (3p-bending) procedure for the derivation of the required results. Also, for the needs of this task INASCO performed all the above mentioned procedures for the selected baseline material.

Unfortunately, the produced carbon fibers from NEWSPEC project was not enough to allow the manufacturing of the second plate and thus INASCO limited its work only to the first plate made of PAN-CFs which was used as a baseline in the design of the winglet. Standard samples were manufactured by RTM (Figure 17, Figure 18) and then tested according to ASTM standards.
LAMBORGHINI AUTOMOBILI was in charge to perform tests based on Building Block Approach (BBA) procedure, largely used in Aeronautic Engineering to validate primary structures in Composite materials. The procedure starts from a Phase 1, with the mechanical characterization on coupons based on ASTM standards, then a Phase 2 is given by an element testing that includes S-shape, L-shape, T-shape components, by increasing geometry complexity.

Both tested materials were Pre-preg with the same resin and weave twill 2x2. The results of NEWSPEC are limited to mechanical tests on the baseline material because, due to technical and technological difficulties within the Project that led to a limited quantity of fibers, no tests were performed on the PE-CF material.

CFRP plates made of Hexcel M47 CFs with laminated layup have been produced, therefore overlapping several plies of the same material with the same orientation. In order to obtain the performances of the materials, it was necessary to produce several plates of different thickness to respect the dimensions of the specimens specified in the test standards. All laminated were placed in a vacuum bag for a minimum period of 1h before the curing cycle. Six plates of dimensions 625 X 600 mm and 2.5 mm thick have been produced, followed by an autoclave vacuum bag curing cycle.

Thermo-physics and mechanical tests were performed on those samples (Figure 19). The tests were performed in L/E-5 department according to relevant standards and conducted successfully unless for the strength compression tests where the results are calculated on less than 5 specimens. The material produced and tested shows slight porosity and delaminations along the thickness. DSC analysis shows that the material achieved a complete cure.

2) Processing of CMC and C/C composites for automotive brakes

Original scope of this task, carried on by BREMBO and PETRO CERAMICS, was manufacturing and testing real scale automotive brakes, in details C/C brake pads, C/C brake rotors and C/SiC brake rotors. However, technical and technological difficulties within the Project has led to a limited quantity of fibers, enough to manufacture only small scale samples (Figure 20, left).

In order to manufacture a C/SiC specimen, first activity is chopping NEWSPEC fibers to the length of current reference fibers used to manufacture a Brembo C/SiC rotor, for instance around 9mm (Figure 20, right). Fibers were chopped to requested size, however chopping procedure was more difficult than reference material due to NEWSPEC fiber high brittleness. This was due to the fact that the supplied PE fibres were not optimized in terms of quality and mechanical performance.

The raw material for a CCM (C/SiC) final material was composed of a blend of fibers and phenolic resin, with optionally the addition of fillers. Different recipes were defined to manufacture different C/SiC coupon types.

Physical and mechanical tests showed that the CCM produced by using the PE-CFs have quite poor mechanical characteristic: MOR and MOE are on average 3 times worse than reference CCM material. Thermal conductivity of CCM-3 sample is in the range of ref material, slightly higher presumably due to higher Si/SiC content.

Similar results were obtained also on C/C sample materials, NEWSPEC C/C final piece has lower mechanical properties than ref C/C material. Poor mechanical properties and big fiber diameter are presumable related.

3) CFRP composites for oil and gas sector applications
The design and technological documentation for the production of the demonstrators by YSDO, including the program for winding the demonstrators, namely elements of oil pipelines (tubes) and high-pressure vessels for natural gas storage, cable bandages from selected carbon fibers, has been developed. Within the framework of the project, new carbon fibers were obtained on the basis of UKN/5000 (T300 type), TC36S (T700 type), IMS65 (T800 type) of commercially available UKN/5000 fibers treated with atmospheric plasma at VITO using acrylic acid and allylamine. The properties of new fibers were determined on samples and it was established that the plasma treatment process is universal and applicable to all types of fibers, including for PE-based fibers.

UKN/5000 carbon fiber treated with plasma with acrylic acid was chosen out of three types of investigated carbon fibers, as it appeared to be the most technological considering composite manufacturing by wet winding technique.

Characteristics of composite material made on the basis of a new carbon fiber obtained by VITO plasma treatments for all selected types of fibers are sufficient for the manufacture of high pressure natural gas storage vessels (Figure 21) and confirm the correctness of the project concept as to plasma influence on carbon fiber.

Results of LCA and LCC analysis
A main goal of NEWSPEC was to assess and possibly quantify the production process performance in terms of Life Cycle Costing and Life Cycle Assessment.

LCC allowed us to make a comparative life cycle cost assessment of our new process compared to conventional PAN-CFs manufacturing. The approach taken is to use public data about a scaled up plant (1,500 ton CF per year) for PAN based carbon fibres and estimate and document the fundamental performance differences that occur when using NEWSPEC technology.

This work covered the production phase: from cradle to gate. The end-of-life phase, which is not presented in this report, was also covered as part of WP8.

Data are collected from the literature, several data gathering meetings at ITCF and TECNARO, inputs from members of the installed partners Task Force for data gathering for WP8 and finally LCA databases.

The NEWSPEC process flow diagram (Figure 22) is based on the input from project partners involved in the technological development of the process.

We only present a very brief summary here of our LCC analysis (Fig. 23-24).

The results of our calculations show a base case of 22,07 €/kg for the conventional PAN based process. Where the main factors contributing to variance are: Carbon yield, Wage rate, Electricity price, Investment in wet spinning production technology and Raw material cost.

The results of the calculations show a base case of 18,96 €/kg for the NEWSPEC process. The usage of Bio PE would increase the precursor cost with 1,50 to 2,60 €/kg CF resulting in a total cost of € 20,46.

Main factors contributing to variance in the result are:
• The carbon yield: this indicates it is important to verify the 73% carbon yield
• The wage rate: for now the labour requirements are assumed to be the same in the NEWSPEC process as in the PAN based process. It should be checked whether there are reasons to assume differences in labour requirements
• Electricity purchase costs have a large impact
• Nitrogen purchase cost: given the large amount of nitrogen usage the different possibilities for nitrogen purchase or production (generation plants) and their associated costs should be considered new to overall optimisation of nitrogen gas usage over the entire process
• Spinning maintenance and other costs: seem high compared to the 3% of other costs estimated in the Harper cost model and should thus be looked at in more detail, when engineering the NEWSPEC process in the future.

When including lower yielding SRUs and abatement technologies the results of the calculations show an estimate for the base case of €16,65/kg for the NEWSPEC process. However, the usage of Bio PE would increase the precursor cost with 1,5 to 2,6 €/kg resulting in a total cost of € 18,15/kg CF. The cost per kg CF of the sulphur recovery and post combustion treatment in the case where the SRU and the abatement technologies are included (€0.78/kg CF) is lower than the assumed €3/kg in the basic NEWSPEC production process, leading to the overall drop in the total cost per kg CF.

The impact of abatement strategies to recover sulphur are accounted for separately in the LCA and LCC. Moreover, given the price fluctuations of petroleum over the lifetime of the project, an additional analysis was added to document the repercussions of the price fluctuation on the prices of the conventional PAN based process and the NEWSPEC process.

The choice for PE and melt-spinning offer significant cost savings to produce carbon fibres compared to the conventional PAN based process where wet spinning is standard practice. The increased use of nitrogen and the higher abatement cost due to the use of sulphur are important additional cost factors of the NEWSPEC process compared to the conventional PAN based process. Applying a scaled up double spot Plasma treatment as surface treatment slightly decreases the costs of the NEWSPEC production process compared to a process with conventional surface treatment. Price fluctuations in petroleum affect the PAN-based process more than the NEWSPEC process if the precursor prices are assumed to evolve according to the price index of imported petroleum.

In Figure 25, a summary of the cost calculations (including a low-high range of +/- 25%) shows that, in the most optimistic scenario, NEWSPEC PE-CFs could range between 12 to 15 €/kg, depending on the various technological choices, which is a bit higher than what we estimated at the beginning of our programme i.e. around 10 €/kg or less.

The LCA shows that the choice for melt spinning of the PE precursor material creates environmental benefits compared to the wet spinning of PAN precursor. PE as precursor material for carbon fibres as itself creates already a reduced environmental impact, as the PE production process has a lower environmental impact than PAN production (mainly due to the impact of the acrylonitrile). The use of sulphur itself during stabilization creates no significant environmental effect, and also the impact of potential remaining emissions of sulphur to the atmosphere (which is limited due to recycling and abatement processes) is not significant.

The LCA also shows the positive effect of replacing the conventional surface treatment by atmospheric plasma treatment, which leads to a reduction of the NEWSPEC process’ environmental impact of on average 5%.

Potential Impact:

POTENTIAL IMPACT

The restrain of climate change reversal, the decrease in energy prices and the energy efficiency are the main drivers for a future sustainable economic growth. It is known, for instance, that fuel saving for terrestrial vehicles or airplanes can help towards deferral of global warming and, thus, can pave the way for a better future for our planet. Carbon fibre reinforced composites (CFRC) are 80% lighter than steel and 50% lighter than aluminium, while delivering at least twice the strength and stiffness. The reduction of the overall weight of an aircraft by 60% can increase considerably its effective payload and can also yield to significant savings in fuel consumption.
In the automotive industry, there is enormous potential, all the way to new designs, to reduce the mass of many relevant components (body, chassis, interior parts and brake systems) and therefore decrease dramatically fuel consumption and GHG emissions. CF cost still represents the main market barrier for the mass-market penetration of those materials, therefore the primary need of the automotive supply-chain industry is to reduce the cost of CF composites.

The use of CFs in wind energy blades is projected to be the first largest application sector by 2020. New offshore and onshore wind power plants with much higher degrees of efficiency based on CFRC will help generate alternative energy economically in the near future. The increasing dimensions of the wind turbine blades (>1MW), especially in offshore areas, will require the extensive use of CFs due to the enormous tensile loads on rotors with large diameters. Retrofitting of already installed (smaller) turbines is also of considerable interest for the “wind companies”. Here low-cost CFs could be an ideal replacement for classical low module E-glass fibres providing enhanced technical performance at competitive cost.

Enormous opportunities for CFRC application is given by civil and industrial infrastructures building. One such promising field is that of oil/gas transportation and storage (compressed natural gas tanks, offshore oil wells, pipelines and gas turbines). The largest new potential end use for CFRC could be the manufacture of riser pipes, drill risers and choke and kill lines for the offshore oil industry: one single 3000 m CFRC riser string with 100 joints consumes about 60 tonnes of CFs, as 17 large wind blades or 200 sport cars today. The future is also likely to witness the growth of CF in the fuel cell industry for the making of natural gas and hydrogen storage systems.

Therefore, given these premises, low cost CFs will open several new application perspectives and, by consequence will have high industrial, environmental and societal implications. In order for Europe to achieve higher independence from imported CFs and fossil oil-based precursors, mainly PAN, it is necessary to invest resources on development of novel strategic CF precursors. This shall bring about novel, technically valid, cheaper and environmentally sustainable materials.

NEWSPEC applied research proposes indeed promising materials, such as poly-olefin, which can be sustainably derived from bio resources (bio-ethanol). The PE-CFs could provide the highest performance/cost ratio than any other precursor currently under investigation. At this PE combines high availability and versatility, since non-fossil alternatives are available at very competitive market prices.

The NEWSPEC Project has generated fundamental know-how related to low-cost CFs manufacturing from innovative precursors, such as polyethylene. Before NEWSPEC, many unknowns were present in the chemistry of PE material and several risks related to its handling were not yet well defined. NEWSPEC made a big step forward in this direction: during the project a huge number of compounds were tested, many diverse solutions were tried and lot of attempts were done. Many of them failed, but all those failures helped us to find the right direction for an original PE-CFs processing.

Importantly, NEWSPEC ends with two strong international patents, of which one related to the innovative process for solid-phase sulfurization of PE precursor and a very innovative equipment, SULFI, to implement this new process on continuous endless precursor fibres.

Innovative and working prototype machines for continuous endless CFs processing have been also designed, developed and tested, in particular: a melt-spinning process equipment capable of semi-industrial manufacturing of PE fibres, a double-spot plasma equipment for CF surface functionalization and a remote Raman probe for in-line monitoring of mechanical features of the produced CFs.

In conclusion, NEWSPEC project has put good and strong basis on which to build up the manufacturing low-cost PE carbon fibres. Almost all the milestones foreseen by the programme have been reached and the research phase, both at lab scale and pilot level, has been successful.
SOCIO-ECONOMIC IMPACT and WIDER SOCIETAL IMPLICATIONS SO FAR
The development of alternative PE-CFs from the proof-of-concept up to mature exploitable technology required a critical amount of knowledge on several aspects: chemistry of precursor and its modification through nano-materials; chemical engineering know-how for the proper development of original processing paths and treatments; computational methods for process optimization; advanced on-line inspection techniques for CFs; expertise in CF characterization, including surfaces and fibre/matrix interfaces; and composite material processing up to product developments and validation.

NEWSPEC, as a Large Scale collaborative project, put together the best knowledge and expertise in related fields from academia/research and industry. This ultimately resulted in new trained researchers and enterprise workforce - in fact specific training activities were included in NEWSPEC - with both direct and indirect benefits from the scientific and social point of view.

NEWSPEC can thus contribute towards FP7 and Horizon2020 objectives to create growth and jobs, especially for the benefit of SMEs.

DISSEMINATION STRATEGY
The objective of the dissemination strategy applied was to identify and organise the activities to be performed in order to maximise the influence of the project and to promote commercial and other exploitation of the project results.

In more detail, the objectives of the dissemination were:

i. To raise public awareness about the project, its expected results and progress within defined target groups using effective communication means and tools;

ii. To exchange experience with projects and groups working in the domain of carbon fibres production in order to join efforts, minimize duplication and maximize potential;

iii. To disseminate the fundamental knowledge, the methodologies and technologies developed during the project;

iv. To pave the way for a successful commercial and non-commercial exploitation of the project outcomes.

The following general subjects of dissemination were identified:

1. NEWSPEC project itself (general scope, coverage, goals and milestones and plans to reach them)
2. interim results (reached objectives and achievements)
3. techniques and methodologies (in respect of IPR issues)
4. technologies (in respect of industrial IPR issues)

According to the Article II.29 of the EC-GA “Each beneficiary shall ensure that the foreground of which it has ownership is disseminated as swiftly as possible.” Therefore, every possible opportunity was embraced by individual partners or on collective basis through joint appearance by more than one partner to make NEWSPEC known among professionals and general public as well.

Partners of the consortium contributed to the dissemination according to their foreseen role and effort and using all available tools, for instance by participating and giving presentations at conferences, publishing papers, networking and similar activities and strived to maximize the existing dissemination channels for the purpose of project result adoption and successful future industrial exploitation of NEWSPEC outputs. Dissemination activities relied on the effort and the possibility of each partner in exploiting opportunities to present the project and its result. Therefore, ensuring effective internal communication and dissemination among the Consortium partners represented an important key success element for the Project. Periodic Meetings and TELCOs and the set-up of a Project Collaborative space (on the ©EMDESK Platform) were
used as means to ensure a constant and effective of internal dissemination. External dissemination targeted Academic and research community, Industrial sector, Industrial Associations, Government bodies and policy makers as well as Media and public community, at national, European and International level. Dissemination activities were planned in accordance with stage of the development in the project as planned in the Description of Work (DoW). Although a number of dissemination activities took place during the first 24 months of the project the most significant dissemination activities took place as intermediate and final research results became available. Dissemination activities in NEWSPEC project were and are deeply wedded with the intellectual property (IP) rights protection which is clearly stated in EC-GA Articles II.26 - 29. Practical application of IP rights protection agreed among NEWSPEC project partners is adjusted in the Consortium Agreement (CA) in Section 9.

DISSEMINATION ACTIVITIES
A common graphic identity was defined at the beginning of the Project to allow for better visibility and recognition as well as branding of the NEWSPEC project. Therefore, all dissemination tools and activities referred to or included: the name of the project: NEWSPEC, the project’s website URL, the NEWSPEC project logo, and the acknowledgements to EC public funds: “The research leading to these results has received funding from the European Union’s Seventh Framework Programme (FP7/2007-2013) under the NEWSPEC project, grant agreement no. 604168.”

Project websites are one of the main communication tools of projects funded under the EU 7th Framework Programme. To ensure maximum visibility to the NEWSPEC objectives and results a project website was set up registering in the “eu” domain and with an intuitive URLs to increase hit rates: http://www.newspec.eu

The design of the website built upon the following criteria and taking into account suggestions given in the EU Project Websites – Best Practice Guidelines (EC, 2010):

i. visual communication: use of colours and/or photos, web pages are easy to browse, information is kept short and links are included to websites, publications, and so on.

ii. verbal communication: the website uses simple phrasing, no jargon is used in order to attract the widest possible audience, e-devices are user friendly.

iii. visibility: maximum use of free or affordable methods to increase page ranking on search engines, Webmaster Tools provided by search engines to check indexing status, good cross-linking between the different pages of your site and other sites, add keywords to the web page metadata; use frequently used keyword search phrases both in the metadata and in the contents pages.

iv. regular update of contents: the website was maintained by WG and the update regularly done by the Webmaster upon inputs from the partners; social media were also used.

v. Monitoring and feedback tools: the website includes a counter of visitors to measure the number of visits; a visitors’ feedback form, to get a feedback on the usability of the web site and on the interest created by the project.

The public section of the NEWSPEC website therefore:
- provides a brief project summary in journalistic style highlighting the objectives, the contents and the structure of the NEWSPEC Project including the composition of the NEWSPEC Consortium.
- provides a short profile of each of the NEWSPEC Partners and a link to the related web sites;
- provides access to the project Public Deliverables and abstracts of selected non-Public Deliverables;
- provides copies of publications and presentations done at external conferences;
- features a separate events section where events are registered and highlighted. It refers to NEWSPEC events such as NEWSPEC meetings and workshop, and conferences and external events where NEWSPEC had an active role (e.g. presentation of paper(s), organisation of sessions, stands with demos, etc.).

Also, Social media channels were activated and maintained to increase visibility and thus raise public awareness:
- Facebook https://www.facebook.com/newspecproject/
- LinkedIn https://www.linkedin.com/groups/7472491
- Twitter https://twitter.com/NewspecProject?lang=it

NEWSPEC standard Flyer and Poster were issued, published, restyled and distributed throughout the project duration, with the aim to intensify and strengthen the effect of recognition of the Project.

The main objective of the project brochure is to provide our audiences with an attractive and written project overview and a summary of the main project objectives and characteristics. To assist the dissemination effort, an attractive and professionally made brochure has been prepared by WG and published on the project website.

The brochure presents the goals of the project and the main (expected) findings. The text is designed taking in to account not only experts, but also an interested non-specialist. It introduces the main mission and the goals of the project. Furthermore, it includes the website address and provides basic information on Consortium. All partners’ logos are also displayed.

The main purpose of the project poster is to catch the audience attention. The posters focuses on the visual aspects. The content of the poster has to be clear and easily understandable by the target end users. To reach this objective an eye- catching poster was designed, then restyled and updated. With regard to the layout and design, the poster shows the project’s logo and the colours emphasizing the link to the project´s graphic. From the content point of view, the poster illustrates the project objectives and includes basic information on the project and on the Consortium, including all partners’ logos. It is possible to download it from the NEWSPEC website.

Press releases on partners’ websites were issued at the beginning of the project. Articles were published on a few scientific journals (CARBON, Journal of Raman Spectroscopy, Composites Science and Technology, Technological systems) and more popular magazines (PlatinumOnLine, OmniaAuto.it) throughout the project duration.

Posters were presented at relevant conferences and the project results presented orally to many congresses and scientific events (such as: JEC Europe, USA and Asia, Industrial Technologies and Euronanoforum, ACS National Meeting and Exposition, Fakuma trade fair, Nanotechnology, Aachen-Dresden International Textile Conference (ADITC), AMI’s Wind Turbine Blade Manufacturer, EMC2 Connecting meeting, INTERNATIONAL CONFERENCE ON BIOBASED TEXTILES AND PLASTICS, WITEC Raman Microscopy Conference, Annual EWEA event, User Forum on Atmospheric Pressure Plasma Applications, GOCARBONFIBERS, etc). Several sector Stakeholder and Academic representatives were contacted to discuss the project perspectives on all occasions.
Also, a workshop on "Development, manufacturing and application of Carbon Fibers and Fibre-based materials: the new frontier for low cost and green processes" was organized in June 2016, during the Industrial Technologies conference. NEWSPEC Consortium met the Carbon Fibres & Advanced High-Performance Composites Cluster representatives, academic and industrial stakeholders and interested parties, to discuss research results and future developments, establishing potential synergies and collaborations to further NEWSPEC research objectives and results in the future.

Training activities were also organised, including two SummerSchools (1st and 2nd European Course on Next Generation Carbon Fibre Processing), with the idea to contribute to the scientific and professional development of researchers and other key staff, research managers, industrial executives, and potential users of the knowledge generated by the project.

EXPLOITATION OF RESULTS
NEWSPEC produced a relevant number of exploitable results which are not only interesting for carbon fibre manufacturing industry but also to other stakeholders such as advanced polymer manufacturers, textile producers, plastic compounds makers, machines manufacturers and various end-users of the carbon composite material chain.
The perspective of this Consortium is to finalized the experimental work at pilot level scale in order to definitively prove the industrial feasibility of our idea. This will include also a testing phase of the PE-CFs in cooperation with carbon composite makers. Once this will be done, it will be necessary to aggregate investments for a process scale-up to a pre-industrial manufacturing line with a limited production capacity (e.g. some tons per year), that are required to fully evaluate the technical and economic viability of this technology. We hope this could be done thanks to public-private partnerships, joint ventures or direct industrial investors and we are already looking forward in this direction.
List of Websites:
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Related documents

[final1-newspec-finalreport-figures-and-tables.pdf]

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