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PROJECT FINAL REPORT

Grant Agreement number:	**ACP7-GA-2008-213267**
Project acronym:	**LAYSA**
Project title:	**MULTIFUNCTIONAL LAYERS FOR SAFER AIRCRAFT COMPOSITE STRUCTURES**
Funding Scheme:	**SMALL OR MEDIUM SCALE FOCUSED RESEARCH PROJECT**
Period covered:	**from 01/09/2008 to 31/12/2011**

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4.1 Final publishable summary report

1. EXECUTIVE SUMMARY

Based on needs to provide an efficient safety and security system for aircraft composite structures, the main objective of LAYSA project has been focused in establishing the scientific and technological basis for the development of a new multifunctional layer with ice / fire protection and health monitoring capacity to be integrated into composite structures.

This has been approached by the achievement of the following objectives:

i) Scientific objectives:

- To design and manufacture novel layer concept with multifunctionality based on nanomaterials, combining next properties:
  - Electrical / thermal conductivity capable of distribute the necessary heat on composite surface to prevent ice formation on its surface in rough fly conditions or remove the already existing one. With respect to current electrothermal system it can be estimated that a weight reduction of 99% and a power consumption reduction of 50% can be achieved.
  - Fire resistance. Reduced flammability (50% of reduction in Heat Release Rate HRR with respect to base composite material).
  - Sensing of temperature and stress by electrical conductivity variation measures.
- Integration, modelling and validation of multifunctional system in novel structural composite materials.

ii) Technological objectives:

- Coupling of the conductivity characteristics of the composite with ice /fire protection and health monitoring systems.
- Development of modelling tools which will facilitate analysis and design of multifunctional layers.
- Manufacturing and validation of composite components with ice/ fire protection, as well as sensing capabilities for real time temperature and damage assessment.

According of the specifications defined in WP1 by the end-users, different nanomaterial formulations and combinations have been prepared and characterized in WP2. Inside WP2 all the needed raw material have been prepared and supplied by UPPA-CANBIO (nanoreinforcement dispersions), ACG (nanodoped resin films) and TECNALIA (buckypapers) in order to produce the selected prototypes (WP3).
On other hand, regarding modelling, also in WP2, the main work carried out has consisted on the prediction of the electro-thermal behaviour, the mechanical response and the sensing performance of a nano-reinforced polymer material system taking into account factors that affect the relationship "structure (at nano-scale)– properties (at macro-scale)" by enforcing the multi-scale modelling principles. Moreover the modelling of the fire burnthrough response of a composite panel has been also considered.

When the most promising materials and processes had been identified, the optimization of the multifunctional concept and manufacturing process started (WP3). The work consisted of manufacturing trials and a subsequent assessment of the results. Identified problems were solved by making the necessary adjustments to the process before additional manufacturing trials were performed. When the optimization work was finished the manufacturing process was validated by the manufacturing of the demonstrators. A lot of work has been done on testing the demonstrators and prototypes according to a test plan and to assess the test results.

In WP4, a technical and commercial evaluation of products has been carried out with the aim to obtain from previous develop to assess the potentialities to transfer to real applications. The diffusion and exploitation aspects are undertaken to address future activities.

2. SUMMARY DESCRIPTION OF PROJECT CONTEXT AND OBJECTIVES

2a) Project Objectives

Based on needs to provide an efficient safety and security system for aircraft composite structures, the main objective of LAYSA project is to establish the scientific and technological basis for the development of a new multifunctional layer with ice / fire protection and health monitoring capacity to be integrated into composite structures. This will be approached by the achievement of next objectives:

i) Scientific objectives:

- To design and manufacture novel layer concept with multifunctionality based on nanomaterials, combining next properties:
    - Electrical / thermal conductivity capable of distribute the necessary heat on composite surface to prevent ice formation on its surface in rough fly conditions or remove the already existing one. With respect to current electrothermal system it can be estimated that a weight reduction of 99% and a power consumption reduction of 50% can be achieved.
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- Integration, modelling and validation of multifunctional system in novel structural composite materials.

ii) Technological objectives:

- Coupling of the conductivity characteristics of the composite with ice /fire protection and health monitoring systems.
- Development of modelling tools which will facilitate analysis and design of multifunctional layers.
- Manufacturing and validation of composite components with ice/ fire protection, as well as sensing capabilities for real time temperature and damage assessment.

Final objective of the LAYSA proposal is to develop a multifunctional layer with thermal and electrical conductivity, improved fire performances and sensing capabilities to be incorporated in aircraft composite structures for ice and fire protection, as well as health monitoring. This can be achieved with synthesis of a nanocomposite layer consisting of a polymeric matrix dopped with fibrous nanomaterials like CNTs (Carbon Nanotubes) or CNFs (Carbon Nanofibres) that, afterward, will be incorporated in the manufacturing process of composite parts.

This can be achieved with synthesis of a nanocomposite layer consisting of a polymeric matrix dopped with fibrous nanomaterials like CNTs (Carbon Nanotubes) or CNFs (Carbon Nanofibres), that, afterward will be incorporated in the manufacturing process of composite parts. The goals of the LAYSA consortium clearly contributes to the two Top-Level Objectives for European aeronautics, identified in the Strategic Research Agenda and the Vision 2020 Report:

1. To meet society's needs for a more efficient, safer and environmentally friendly air transport.
2. To achieve global leadership for European aeronautics, with a competitive supply chain capable of exploiting all of the expertise in Europe, turning new technologies into competitive products.

Furthermore, the proposed work clearly addresses the requirements of Work Programme 2007, Cooperation, Theme 7 TRANSPORT (including AERONAUTICS), Sub-theme AERONAUTICS and AIR TRANSPORT of the 7th Framework Programme.

AREA: 7.1.3.3 Aircraft Safety. AAT.2007.3.3.2 Systems and Equipment

As stated in B1, due to organic nature of polymeric matrix component, composite materials are electrically and thermally bad conductors and they tend to burn easily, emitting toxic gases and smoke. For that, they require affordable, effective and certifiable protection systems against atmospheric hazards such as icing, as well as fire and burning not only for accidents prevention, but also for survivability in case of them.

AAT.2007.3.3.2 Systems and Equipment	
Objective	**How**
-Advanced technologies for aircraft protection against icing. **-Advanced concepts for fault tolerant systems.** **-Design techniques and concept for improved fire, heat and smoke protection.**	This will be achieved by a new **multifunctional layer** to be integrated into composite structures **with ice / fire protection and health monitoring capacity**, which can replace the separated systems used currently for that. **Using electro thermal conductivity and sensing capacity of nanomaterials**, a multifunctional system could be integrated in the composite structures, that: • **It does not have any detrimental effect on the structure and does not suffer by fatigue related problems.** • **No aerodynamic performance penalties** • **Feasible to use for the entire aircraft composite surface.** • **Improved fire protection.**

AREA: 7.1.3.4 Operational Safety. AAT.2007.3.4.2 Maintenance

The aim of this topic is to ensure that aviation safety at current high standards through the increased enhancement of the safety in air transport operations to reduce accident rate by a80% and to achieve a substantial improvement in the elimination of and recovery from human error.

The continuous assessment of remaining life of aerospace components at every stage of aircraft service life remains critical in order to ensure its structural integrity and service capacity. Moreover, improved in field inspection techniques for continuous assessment of their structural health are required with the increased use of composite materials. Current technologies address those aspects separately.

AAT.2007.3.4.2 Maintenance	
Objective	**How**
-Continuous health and usage monitoring; smart maintenance systems including self-inspection.	This will be achieved by replacing the current embedded sensors, by a unique **nanocomposite layer with inherent sensing capabilities:** • Current intense inspection and maintenance of heating elements and fire protection coatings will be avoided, and on the other hand it will be possible a continuous health and usage monitoring and self-inspection; summarizing, **a "smart maintenance" will be achieved.**

AREA: 7.1.4.2 Aircraft Operational Cost. AAT.2007.4.4.2 Aerostructures

The aim of this topic is to ensure cost efficiency in air transport focussing on the reduction of aircraft direct operation costs. Research work will address a wide range of concepts, innovative solutions and technologies which will reduce weight, fuel consumption, maintenance and crew operational costs as main contributors. In AAT.2007.4.4.2, the development of advanced concepts and technologies for increased and optimized use of light-weight composite materials as well, advanced concepts for increased integration of additional functions, as sensing, are looking for wider applications at los cost and weight.

Gaining this knowledge will enable the EU aeronautical industry to respond to the need for economical aircraft and to be more competitive in the world market. Aeronautics industry is important constituent of the sustainable growth of Europe and operates in a highly competitive and dynamic environment. Competition mainly comes from the strong position of US industry, and this fact gives a continuous challenge to proceed in cost effectiveness.

Along these lines, the aeronautic industries are steadily increasing the percentage of composite materials in the structure of the aircraft in order to reduce weight and number of parts. This target coincides with the use of multifunctional materials.

To achieve the objective of increasing the competitiveness in aerospace manufacturing industry, the developments within the LAYSA project will introduce a multidisciplinary approach for the realization of aerospace materials and structures with multifuctionality, in terms of ice / fire protection and sensing capacities, by the use of nanocomposite technology. Nanomaterials can combine excellent electrical and thermal conductivity with unique sensing capabilities. As a results, important weight and maintenance operation reductions can be achieved:

AAT.2007.4.4.2 Aerostructures	
Objective	**How**
-Aerodynamics. Concepts, technologies and systems for drag reduction. **-Structural weight reduction. Advanced structural concepts for increased and optimized use of new composite materials in primary structures; concepts, technologies and systems for application of “smart” materials, nano-technologies, and realization of “smart structures”.**	This will be achieved by replacing the current embedded sensors, heating elements and fire protection coatings by a unique nanocomposite layer with inherent sensing capabilities: • Structure **manufacturing operations will be reduced** considerably. • **No aerodynamic performance penalties** • The current heavy elements for ice protection (foils, wires, meshes, etc) and fire protection (fillers) that increase considerably the weight of the structure, by a unique **lightweight nanocomposite layer** that will be integrated in the composite structure (**99% of weight reduction is estimated**). • Power required for icing protection will be also considerably reduced (**50% of power reduction is estimated**), and as a consecuence generator weight reduction.

2b) Distribution of Work

Based on needs to provide an efficient safety and security system for aircraft composite structures, the main objective of LAYSA project is to establish the scientific and technological basis for the development of a new multifunctional layer with ice / fire protection and health monitoring capacity to be integrated into composite structures.

This will be achieved in two main steps:

1. <u>LEVEL 1:</u> Synthesis of a nanocomposite layer with tailored properties (electrical / thermal conductivity, fire resistance and sensing capabilities), consisting of a polymeric matrix dopped with fibrous nanomaterials.
2. <u>LEVEL 2:</u> Incorporation of nanocomposite layer in the manufacturing process of composite demonstrator.

This project is composed by 12 organisations and it has been constituted to gather the complementary competencies required to reach its objectives:

- **Raw material manufacturers** (HUNTSMAN, ADVANCED COMPOSITE GROUP) for the composite industry that will work in the procurement of formulated products to synthesize and Incorporate nanocomposite layer in composite structures, by selection and modification of resins for LEVEL 1, and layer concept (to be defined: prepreg or gel-coat or resin film,…) for LEVEL 2.
- **Research organisations** (TECNALIA, SICOMP, CNRS-CRPP, UNIVERSITY OF PATRAS, UNIVERSITY OF LILLE, UNIVERSITY OF CRANFIELD, UNIVERSITY OF PAU-CANBIO) that will lead the project and bring essential theoretical background about synthesis, characterization and modelling of nanocomposites and composites, as well as icing technologies.
- **Technology supplier to aircraft end-users** (INASCO, ARIES COMPLEX) that will work in monitoring of multifunctional properties to assure the provision of developed technology to end-user,
- **Aeronautical end-user** (AERNNOVA) that will validate the technology and ensure its exploitation.

Participant	Country	Profile	Key Expertise	Role in the Project
Fundación INASMET (TEC-INAS)	Spain	RES	Private R&D Institute focused on materials	Coordinator. Synthesis and characterization of nanocomposite with electrical / thermal conductivity.
SICOMP AB (SICOMP)	Sweden	RES	Private R&D Institute focused on polymeric composites	Incorporation of multifuctionality in novel nanocomposite. Process development and testing.
Centre de Recherche Paul Pascal (CNRS-CRPP)	France	RES	Public Research Institute focused on materials, soft condensed matter and nanotechnologies	Nanomaterials previous treatment and characterization for electrical / thermal conductivity and sensing capabilities.
University of Patras (UP)	Greece	HES	Applied Mechanics laboratory, focused on materials and structures	Nanocomposite modelling and characterizaton
University of Lille (UOLI)	France	HES	Chemistry and chemical engineering laboratory focused on flame retardancy of polymeric materials	Synthesis and characterization of nanocomposite with fire protection performance
Cranfield University (CU)	United Kingdom	HE	Icing, Droplet impact, Erosion	Examine the dynamic water filming and icing behaviour of the nanocomposite surfaces and how these is affected by erosion
INASCO (INASCO)	Greece	SME	Private SME company focused on aeronautics, advanced materials and manufacturing, and technology design tools	Monitoring and characterization of nanocomposite with electrical /thermal conductivity and sensing capabilities
UNIVERSITY OF PAU-CANBIO (UPPA)	France	HES	Advanced composites based on nanofilers and biofilers. Chemistry on and from nanofilers surface	Synthesis and characterization of carbon nanotubes with high dispersion quality and controlled interface using controlled radical polymerization.
ADVANCED COMPOSITES GROUP (ACG)	United Kingdom	IND	Prepreg manufacturer	Incorporation of technology into matrix products, characterization and evaluation of performance.
HUNTSMAN (HUN)	Switzerland	IND	Epoxy resin manufacturer	Procurement and characterization of appropriate epoxy resin for formulated material.
ARIES COMPLEX (ARICOM)	Spain	IND	Experience and Innovation for new demands in aerospace services	Full services for the design, manufacturing, assembly and product support of structures and interiors
AERNNOVA (AES)	Spain	IND	Aerospace manufacturer	Definition of structure requirements, testing and validation of technology

Table 1 summarizes the profile of each partner of LAYSA Consortium.

3. DESCRIPTION OF THE MAIN S&T RESULTS/FOREGROUNDS

WP0 (Project Management)
-Creation of the website of the project www.laysa.eu with the update information of the project. The website is divided in two different areas, one for public dissemination of the project and, the second one, only for project members.
-Organization of progress project meetings (KOM, 4 months, 9 months, 12 months and 18 months meetings)
-Organization of several technical meetings (at CANBIO and CNRS-CRPP facilities)
-Creation of the project members contact list. (See attached Annex A)
-Organization of progress project meetings (24 months, 30 months, 36 months and final (40M) meetings)
-Organization of several technical teleconferences (CANBIO, ACG, SICOMP)
-Updating of the project members contact list. (See attached Annex A)
-Preparation of the Amendment to the Contract (July 2011)
-Preparation of the official reporting documents.
-Distribution of the funding between partners.
-Coordination of the diffusion actions in different forums (Aeronautics Days 2011, Matcomp 2011, JEC Composites Show 2012)

WP1 (Definition of requirements and materials selection)
T1.1: Our aim is to investigate and demonstrate the capacity of a material system featuring nano-particle doping, to provide functional and structural capability for the skin or an aircraft. This draws together activities which have traditionally been dealt with separately as materials and as systems contributions. In this task, we gather together concepts and data from these two disciplines in order to establish our terms of reference and our specific technical goals. Specifically, the material properties sought are considered in terms of the multiple requirements pertaining to a service aircraft.
T1.2: Based on materials identified in T1.1, screening and identification of the most suitable materials and processes for nanomaterial inclusion in epoxy resin at LEVEL 1 have been carried out. For that, preliminary manufacturing trials have been made, where parameters to evaluate have been on one hand the success in dispersion of nanomaterials in resin (key factor), and on the other hand electrical conductivity and fire behaviour of processed nanocomposite. Testing plan has included: structural characterization (SEM, TEM, Optical Microscopy), thermal characterization (DSC), mechanical characterization (flexural), electrical characterization (electrical conductivity) and fire behaviour (TGA, mass – loss calorimeter). Based on specifications of T1.1 and testing results, the most suitable material candidates have been selected to be used during the following tasks.

WP2 (Synthesis of multifunctional nanocomposite layer, LEVEL 1)

WP 2.1:<u>Development of Electrical/Thermal Conductivity</u>
Raw carbon nanotubes are under the form of a light and disordered powder. For several applications more compact and ordered forms are needed. In particular ice protection devices that operate through Joule's heating have ideally to be made of almost pure nanotube arrays or of composites with a large fraction of nanotubes.
Important work have done on the process parameters for the production of 1.5 and 2 wt.% CNT **masterbatches** based on epoxy resins. But direct loading of polymer matrices is a difficult approach for achieving large volume fractions of nanotubes. Viscosity increase and the presence of aggregates make a direct processing difficult. **Other approaches** are investigated in LAYSA with materials under the forms of **buckypapers** or **fibres.** Such forms are obtained from aqueous and homogeneous dispersions of nanotubes. These dispersions are then either membrane filtered or coagulated to obtain structures with a large fraction of nanotubes. Partners (Tec-INAS, UPPA-CANBIO and CRPP) contributions consisting in preparation and characterizations of these materials in order to selected the best option**. Regarding electrical/thermal conductivity, buckypaper approach reaches the objectives defined for this functionality.**

<u>WP2.2: Development of Fire Resistance</u>
T2.2.1: Based on developments in WP1, it was decided to study the effect of nanoparticles on the fire resistance of composites without previous treatment. Innovative concepts based on gelcoats and alternative resins were proposed. Incorporation of nanoparticles in gelcoats has a **limited effect** on the reaction to fire. Use of **alternative resins** such as **benzoxazine** and **IEP22** is a **promising way**.
T2.2.2: Processes for the synthesis of different composites were set up. Samples corresponding to various concepts were manufactured. **Nanomodified gelcoats**, **carbon or glass-fiber reinforced composites**, resins containing **nanoparticles** and **model resins** containing micrometric and/or nanometric fillers were investigated.

The rheological and mechanical behaviour was also characterized for some of them. Due to difficulties in the resin manufacturing, a limited number of samples were produced.

T2.2.3: The fire resistance of nanomodified gelcoats, resins containing nanoparticles and model resins containing micrometric and/or nanometric fillers was tested. Even if the behaviour of the final composite will be different from the one of the samples tested in WP2, due to expected interactions between the different layers, useful conclusions for the rest of the project can be drawn:

- For the development of a fire-resistant epoxy, sufficient **level of dispersion** of carbon nanotubes and montmorillonite in the resin is difficult to achieve.
- The **use of alternative resins** such as benzoxazine and IEP22 is valuable: the combination between good fire retardant properties and another functionality (electrical/thermal conductivity or monitoring) may be reached in the same layer.
- **Ammonium polyphosphate** and different types of **polyhedral oligomeric silsesquioxanes** provide good fire properties to the model resin. **The 50% pHRR decrease expected by the Consortium is reached with this system.**

<u>WP2.3 Development of Sensing Performance</u>

The main objective of this workpackage is to design and process of adequate nanocomposite with sensing capacity that has the capacity monitor temperature and strain concentrations.The task is delayed mainly due to the lack of materials for testing. Different materialssome new materials arrived at INASCO for characterization (CNT fibres from CNRS-CRPP and films from ACG)

Several specimens, having different manufacturing history and material composition, were tested in INASCO and the conclusion we reached are stated below:

- Even though, some specimens had very low resistance, their sensing capability under strain 1% was measurable.
- In almost every test, specimen’s resistance does not regain its initial value, but returns to a greater one after the end of loading. (hysteresis loop)
- Specimens with resistance in the order of kilo Ohm (10^3) exhibit greater maximum resistance values under the same strain percentage than the ones having a resistance in the order of Mega Ohm (10^6).
- Almost linear correlation between resistance and strain is observed in all tests.
- Best data results (high resistivity, high sensing capability and small hysteresis loop) are observed for specimen 31_A10_MINAS2_01_SP1, manufactured at INASMET and specimen GC_32_D_01 manufactured at HUNTSMAN. It should be mentioned that the latter one is the reference sample (rengel with MY0510) for all gelcoat samples without emboding any nanomaterial.
- Although only three cycles loading test was accomplished and low resistance was measured, specimen GC_32_CL2_D_01 also presented good results, as there was no hysteresis loop and ΔR had a respective value.

<u>WP2.4 Modelling Analysis</u>

Taking into account firstly the fact that the modelling analysis has to run in parallel with the others sub-workpackages as well as the variety of the suggested material systems for development in the initiation of the project the investigated material system here was selected independently on this parameter. So considering that the analytically investigated materials system has to follow the standards of a typical aeronautical material a "CNT-doped Polymer Matrix/Carbon Laminated" material system was determined. As a matrix a typical aeronautic Epoxy resin was selected while the tested type of CNTs was the multi wall. Finally as for the structure and the material properties of the Carbon reinforcement is still under discussion while the decision for the integration way of the developed multifunctional polymer in the laminated structure has not been accomplished up to now.

SIGNIFICANT RESULTS:

A) Electrical Model

In the nano-scale modelling level:

- The MFM approaches with high accuracy the experimentally obtained values of the electrical conductivity when the “percolation” parameters are known.
- The pass to the FEM modelling methods leads to the prediction of the “percolation” curve after a concise parametric analysis. The achieved “percolation” threshold is lower than that was arisen from the experimental values. The discrepancy can be attributed to the fact the developed model corresponds to a ideal CNTs dispersion in the polymer matrix. Another reason may be the different admissions that took place in the calculation process between the analytical and experimental results.

B) Thermal Model

In the nano-scale modelling level:

- The orientation of CNTs in the 1D aligned and 2D Random Dispersed (RD) arrangement can contribute significant to the material system thermal conductivity only in the plain level.
- Higher CNTs aspect ratio can lead to higher thermal conductivity values.
- The influence of the CNTs content is moderate for contents lower than 1% per volume. Nevertheless the observed increase is in agreement with the scientific literature’s values. Up to this value an abrupt increase is observed.
- The consideration that an inter-phase formation takes place between the polymer matrix and the CNTs seems to contribute only in high CNTs content.
- Generally it can be said that the MFM approach can give a first accurate sense of the thermal response of the material system.

C) Fire Resistance

- Different experiments were conducted in order to get parameters for fire resistance modelling. High heating rate thermogravimetric analyses helped the modelling of thermal degradation of samples containing nanoparticles and carbon-fiber reinforced composites.
- The main conclusion is that the incorporation of nanoparticles in the different matrixes did not affect the thermal decomposition. Heat capacity was measured by modulated DSC from -50°C up to 200°C. Methods are under development in order to get heat capacity values at high temperatures.
- The decomposition kinetic parameters were used into the development of a fire degradation material model that can be used in numerical models. The material model can be implemented in any FE code either commercial or custom and using the appropriate fire loading and cooling conditions the fire response of a composite structure can be evaluated.
- The developed models using the ANSYS 13 were able to predict the fire burnthrough response of the specimens tested.

D) Sensing Performance

- The developed Electrical and Thermal models in T2.4.1 in all modelling scales can be used for the electro/ thermal response of the not deformed “material form” while the one task overlaps the other at this point.
- Generally observing the nature of the problems that have to be solved in both tasks (“Modelling of Electro-Thermal Conductivity” and “Modelling of Sensing performance”) it is obvious that they are possible to be faced as one modelling problem for the investigation of the electro-thermal-mechanical material’s performance.
- The nano-scale modelling level the Electrical/ Thermal Model for the not deformed “material form” is still in progress while significant results have been achieved for both models. For the development of the deformed “material form” model the completion of the deformed Mechanical model is demanded.
- In the micro- & macro- scale modelling levels the current status can be considered the same with that in T2.4.1.

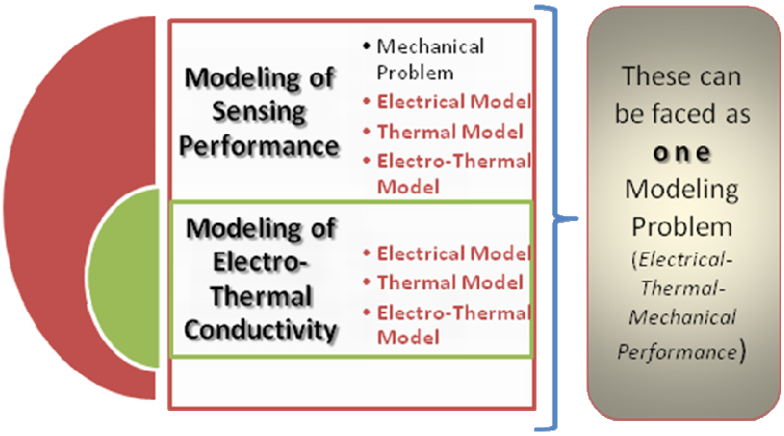


Figure 1. Scheme that declares the overlap between the "Modelling of Sensing Performance" (T2.4.3) and the "Modelling of Electro-Thermal Conductivity" (T2.4.1).

WP2.5. Integration of Functionalities

The three defined functionalities have been afforded separately. Their integration in the final part is closed linked to the processing technology of the part. Due to this subtask will run in parallel with WP3.1, and we can consider that will be included inside WP3.1.

Different concepts of the multilayer configuration have been proposed and, in function of the processing technology (wet or dry processes) requirements and limitations, the best one will be selected in order to reach the objectives of the project.

WP3 (Incorporation of functionality in novel composites, LEVEL 2)

Based on the general concept described in WP1, WP2 and the know-how of the consortium, two possible processing alternatives have been identified:

Wet process

This process is based on infusion technology as the laminate manufacturing process. The insulating layer (thermal and fire protection) will be integrated into the own infusion resin, that is, the fire properties and thermal insulation capability will be developed on the infusion resin; the electro-thermal conductive intermediate layer (heating element and sensing capability) will be formulated as a gel-coat to be applied on the mould surface previously to the infusion process. All the necessary auxiliary elements (thermocouple, connections to electric current source, electrodes, etc) will be also included into this layer. Finally, the thermally conductive and electrically insulating outer layer will be applied on the finished laminate as a coat (top coat or paint).

Dry process

Dry process is based on prepreg technology as the laminate manufacturing process. In this case the insulating layer (thermal and fire protection) will be integrated into the own prepreg resin; depending on the properties achieved, all the prepreg layers or only some of them will be based on the developed fire resistant resin. The electro-thermal conductive intermediate layer (heating element and sensing capability) will be formulated as another prepreg layer or a doped resin film to be included on top of the previous prepregs. All the necessary auxiliary elements (thermocouple, connections to electric current source, electrodes, etc) will be also included into this layer. Finally, the thermally conductive and electrically insulating outer layer will be applied on the finished laminate as a coat (top coat or paint).

The dry process has finally selected to integrate the multifunctional layer developed in earlier work packages in a traditional composite structures manufacturing processes and manufacturing of demonstrator prototypes with multi-functionality incorporated.

A schematic view of the first multifunctional concept proposed for LAYSA project can be seen in Figure 2.

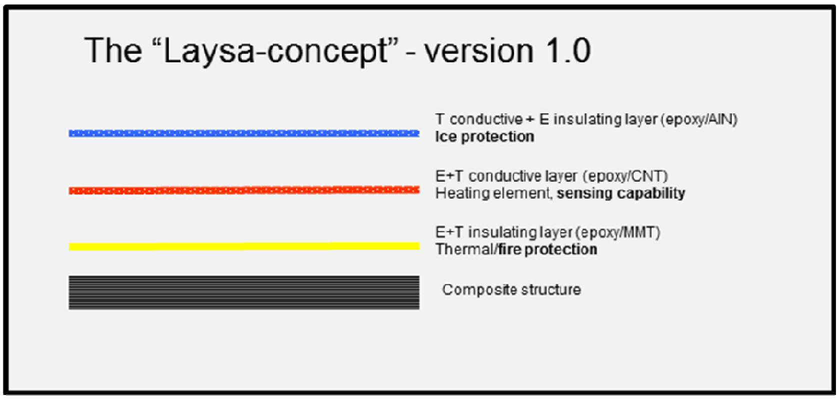


Figure 2: Multifunctional Laysa concept

The work performed by the project partners in this work package has resulted in a recommended concept (Figure 3) and a recommended manufacturing process.

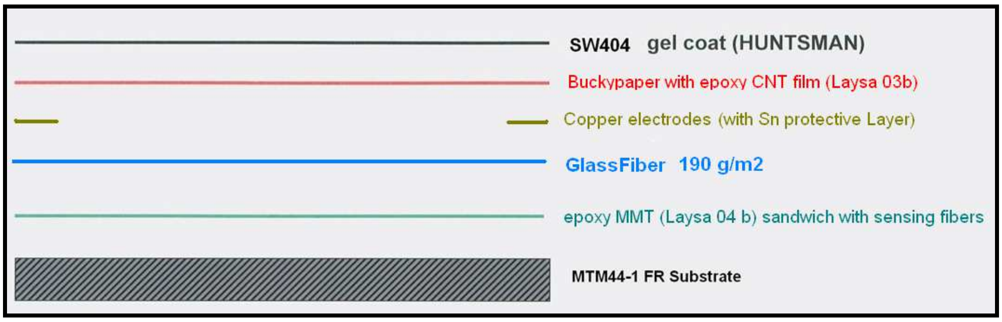


Figure 3: Final concept of LAYSA Project.

Traditional autoclave-cured prepreg manufacturing process has been selected in previous task as the most suitable alternative to develop the required demonstrators.
During the work several prototypes and materials were manufactured and the protypes also underwent different tests. Several processing trials were also performed to optimize process and the properties of the produced multifunctional composite.
In the project several prototypes were manufactured. Some of them were manufactured as a step in the development of proper manufacturing processes and some to pre-test certain properties. To test the final concepts and manufacturing processes a number of final prototypes and demonstrators have been manufactured (Table 2).

Table 2: Specimens manufactured in LAYSA project to test different functionalities.

Reference number	Purpose	Manufacturer	Comments
Sicomp_NA_fire_1	Fire testing	SICOMP	Prototype has fire resistance functionality. *Sent to ENSCL.*
Sicomp_NA_fire_2	Fire testing	SICOMP	Prototype has fire resistance functionality. *Sent to ENSCL.*
Sicomp_NA_fire_3	Fire testing	SICOMP	Prototype has fire resistance functionality. *Sent to ENSCL.*
Sicomp_ref_fire_1	Fire testing	SICOMP	Prototype has no functionality. Used as reference for fire testing. *Sent to ENSCL.*
Sicomp_ref_fire_2	Fire testing	SICOMP	Prototype has no functionality. Used as reference for fire testing. *Sent to ENSCL.*

Sicomp_ref_fire_3	Fire testing	SICOMP	Prototype has no functionality. Used as reference for fire testing. *Sent to ENSCL.*
Sicomp_NA_ice_1	Ice-adhesion testing	SICOMP	Prototype has three functionalities (fire, heating and sensing). *Sent to CU.*
Sicomp_NA_ice_2	Ice-adhesion testing	SICOMP	Prototype has three functionalities (fire, heating and sensing). *Sent to CU.*
Sicomp_NA_sensing_1	Sensing testing	SICOMP	Prototype has three functionalities (fire, heating and sensing). Sensing fibers was covered with resin during manufacturing and thus prototypes could not be used for sensing tests. *Not sent to partners.*
Sicomp_NA_sensing_2	Sensing testing	SICOMP	Prototype has three functionalities (fire, heating and sensing). Sensing fibers was covered with resin during manufacturing and thus prototypes could not be used for sensing tests. *Not sent to partners.*
Sicomp_NA_sensing_3	Sensing testing	SICOMP	Prototype has three functionalities (fire, heating and sensing). Sensing fibers was covered with resin during manufacturing and thus prototypes could not be used for sensing tests. *Not sent to partners.*
Sicomp_NA_heating_1	Heating testing	SICOMP	Prototype has only heating functionality. Main purpose to test type of connector. *Not sent to partners.Evaluation done at SICOMP.*
Sicomp_NA_heating_2	Heating testing	SICOMP	Prototype has only heating functionality. Main purpose was to test type of connector. *Not sent to partners. Evaluation done at SICOMP.*
Sicomp_NA_heating_3	Heating testing	SICOMP	Prototype has heating and fire functionality. Main purpose was to test geometry of heating layer. *Not sent to partners. Evaluation done at SICOMP*

Sicomp_NA_heating_4	Heating testing	SICOMP	Prototype has heating and fire functionality. Main purpose was to test geometry of heating layer. *Not sent to partners. Evaluation done at SICOMP*
Tecnalia_NA_fire_1	Fire testing	TECNALIA	Prototype has ice adhesion and fire resistance functionality. *1st batch, sent to ENSCL on 26 May.*
Tecnalia_NA_fire_2	Fire testing	TECNALIA	Prototype has ice adhesion and fire resistance functionality. *1st batch, sent to ENSCL on 26 May.*
Tecnalia_NA_fire_3	Fire testing	TECNALIA	Prototype has ice adhesion and fire resistance functionality. *1st batch, sent to ENSCL on 26 May.*
Tecnalia_NA_fire_4	Fire testing	TECNALIA	Prototype has three functionalities (ice adhesion, fire and heating). *2nd batch, sent to ENSCL on 4 November.*
Tecnalia_NA_fire_5	Fire testing	TECNALIA	Prototype has three functionalities (ice adhesion, fire and heating). *2nd batch, sent to ENSCL on 4 November.*
Tecnalia_NA_fire_6	Fire testing	TECNALIA	Prototype has three functionalities (ice adhesion, fire and heating). *2nd batch, sent to ENSCL on 4 November.*
Tecnalia_ref_fire_1	Fire testing	TECNALIA	Prototype has only ice adhesion functionality. Used as reference for fire testing. *2nd batch, sent to ENSCL on 4 November.*
Tecnalia_ref_fire_2	Fire testing	TECNALIA	Prototype has only ice adhesion functionality. Used as reference for fire testing. *2nd batch, sent to ENSCL on 4 November.*
Tecnalia_ref_fire_3	Fire testing	TECNALIA	Prototype has only ice adhesion functionality. Used as reference for fire testing. *2nd batch, sent to ENSCL on 4 November.*
Tecnalia_NA_ice_1	Ice-adhesion testing	TECNALIA	Prototype has ice adhesion and fire resistance functionality. *1st batch, sent to CU on 26 May.*
Tecnalia_NA_ice_2	Ice-adhesion	TECNALIA	Prototype has ice adhesion

	testing		and fire resistance functionality. *1st batch, sent to CU on 26 May.*
Tecnalia_NA_heating _ice_1	Heating and Ice-adhesion testing	TECNALIA	Prototype has four functionalities (ice adhesion, fire, sensing and heating). *2nd batch, sent to CU on 17 November.*
Tecnalia_NA_heating _sensing_1	Heating and Sensing testing	TECNALIA	Prototype has four functionalities (ice adhesion, fire, sensing and heating). *1st batch, sent to INASCO on 22 June.*
Tecnalia_NA_heating _sensing_2	Heating and Sensing testing	TECNALIA	Prototype has four functionalities (ice adhesion, fire, sensing and heating). *1st batch, sent to INASCO on 22 June.*
Tecnalia_NA _sensing_1	Sensing testing	TECNALIA	Prototype has four functionalities (ice adhesion, fire, sensing and heating). *2nd batch, sent to INASCO on 4 November.*
Tecnalia_NA_heating _1	Heating testing	TECNALIA	Prototype has only heating functionality. *Trials carried out in Tecnalia, specimens not sent to partners.*
Tecnalia_NA_heating _2	Heating testing	TECNALIA	Prototype has only heating functionality. *Trials carried out in Tecnalia, specimens not sent to partners.*
Tecnalia_NA_heating _3	Heating testing	TECNALIA	Prototype has only heating functionality. *Trials carried out in Tecnalia, specimens not sent to partners.*
Tecnalia_NA_heating _4	Heating testing	TECNALIA	Prototype has only heating functionality. *Trials carried out in Tecnalia, specimens not sent to partners.*
Tecnalia_NA_heating _5	Heating testing	TECNALIA	Prototype has only heating functionality. *Trials carried out in Tecnalia, specimens not sent to partners.*
Tecnalia_NA_heating _6	Heating testing	TECNALIA	Prototype has three functionalities (fire, sensing and heating). *2nd batch, manufactured and tested in Tecnalia.*

ARICOM_Tecnalia_NA_heating _1	Heating testing *Mechanical testing to be carried out by Aernnova?*	ARICOM-TECNALIA	Demostrator, with two functionalities (fire and heating).
ARICOM_Tecnalia_NA_heating _2	Heating testing *Mechanical testing to be carried out by Aernnova?*	ARICOM-TECNALIA	Demostrator, with two functionalities (fire and heating).

To ensure that all necessary aspects of the multifunctional capacities in real conditions are properly evaluated, a test plan was developed. The test plan is presented in this document. Since the partner with the best expertise in each area was responsible for that particular testing it was decided that the detailed plans for each test are delegated to that partner.

The test plan that was decided can be seen in Table 3 below. Details on specific tests are delegated to the responsible partners

Table 3. Test plan used for testing. Details on specific tests are delegated to responsible partner.

Test	Responsible partner	Other contributing partners
Testing of ice protection capacity part 1: Heating functionality	TECNALIA	CU, SICOMP
Testing of ice protection capacity part 2: Ice adhesion	CU	
Testing of fire properties	ENSCL	
Simulation of mechanical properties	UP	
Testing of sensing capacity	INASCO	

An extensive pre-project work was performed to produce the initial project plan. One result from this work was that the following functionalities and properties were identified as probable to test:

1. Ice protection capacity
2. Fire resistance
3. Sensing capability
4. Mechanical properties and composite / nanocomposite adhesion

During the project the consortium decided to omit the mechanical testing and the testing of composite/nanocomposite adhesion. This has to do with the fact that the multifunctional layers were decided to be applied at the top of the bulk composite and thus they would not have to support the mechanical property of the underlying composite. The project has instead produced a very rigorous computational study of the mechanical properties.

At the end of the work package, two leading edge demonstrators were successfully manufactured at TECNALIA-facilities by people from ARICOM and TECNALIA. A leading edge is that part of the wing (or airfoil) that first contacts the air. Heating tests were also performed on the demonstrators with promising results (Figures, 4, 5 and 6). Given the limited functional results obtained for multifunctional specimens when the three functionalities are included, the consortium decided to develop demonstrators including only one or two functionalities. This should allow obtaining clear conclusions of the selected functional layer behaviour when it undergoes real manufacturing cycles and applications.

Due to the good results obtained in previous tasks of this project from the study of the Bucky Paper as de-icing layer when individually tested, and the incomplete results regarding sensing and fire protection at the moment of the demonstrator definition, a leading edge specimen only with de-icing capabilities was selected as a representative aeronautical item of high interest.

The final layer configuration was then easily frozen, based on ARIES COMPLEX large experience in this sector and component, and the design and manufacturing of the mould fulfilled.
Being moulds and all the different materials available, demonstrators have been manufactured modifying layer configuration and some manufacturing parameters for each new manufacturing cycle in order to improve the demonstrator final integrity and test functionality under different manufacturing conditions.

At the moment of the final demonstrator definition, the work performed to develop the multifunctional layer stack including sensing fibres, fire protection and de-icing capabilities onto a CNT substrate had demonstrated no success in the heating test.

Since sensing fibres were not yet optimised (a new batch was pending of characterization), some fire test were also pending and previous test of each individual functionality had provided good results, it was decided to define the final demonstrator on the basis of the Montmorillonite substrate and the Bucky Paper heating capabilities previously demonstrated.
Anti-icing and de-icing technologies are intensively being investigated in the aircraft frame in order to optimize current solutions to avoid effects of ice accretion on an aircraft that may lead to a complete loss of control and/or insufficient lift to keep the aircraft airborne due to accumulation in airfoils and flight control surfaces.

Given the experience of ARIES COMPLEX in manufacturing composite leading edge for different flight control surfaces (wings and stabilizers), and the importance of avoiding ice accretion on such part to assure flight performances, a leading edge demonstrator has been considered as an optimal choice for demonstrating de-icing capabilities of the developed Bucky Paper within LAYSA.

The proposal is to produce a reduced part of a leading edge (400 mm long) which will include the overall curvature. This will allow testing

- the handling and difficulties of adding this functional layer during a typical manufacturing process of an aeronautical part (lay-up, moulding and demoulding).
- how this new layer is adapted to curved parts.
- and the demonstrator integrity after curing: defects (delaminations, porous, etc) and functionality.

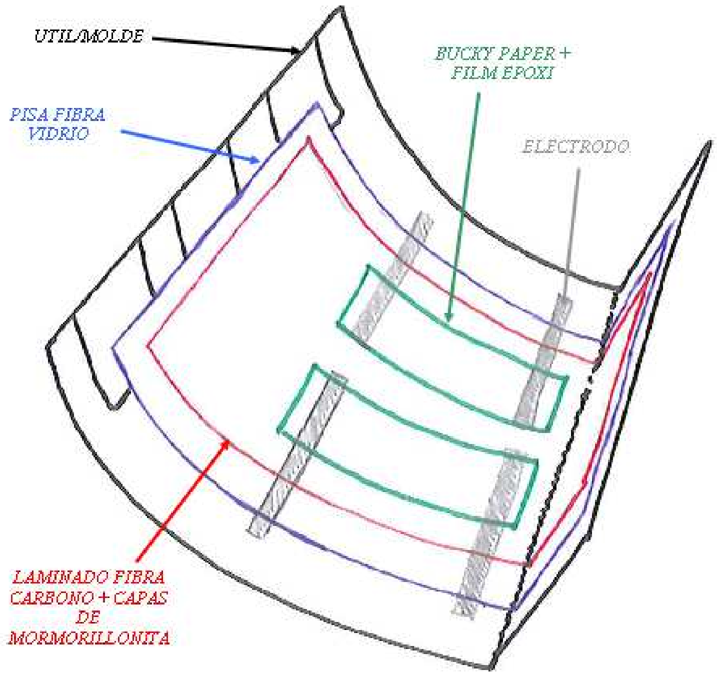
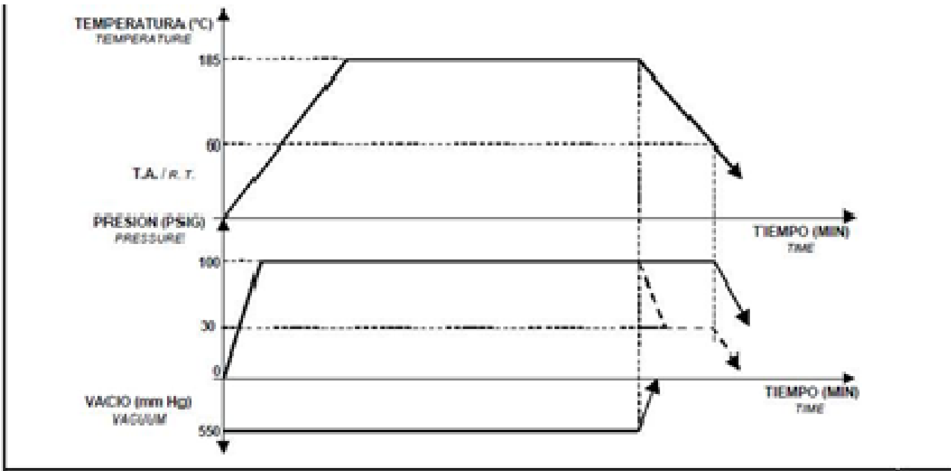


Figure 4 . Overall configuration for the demonstrator.



PARAMETROS/ *PARAMETERS*		NOTA *NOTE*
PRESION / *PRESSURE* (PSIG)	100 ± 5	2
VACIO / *VACUUM* (mm Hg)	550 a 600	1
TIEMPO DE CALENTAMIENTO / *HEATING TIME* (MINUTOS)	82 a 410	3
TEMPERATURA DE ESTABILIZACIÓN / *STABILISED TEMPERATURE* (ºC)	185 ± 5	
TIEMPO DE ESTABILIZACIÓN / *STABILISED TIME* (MINUTOS)	120 a 180	
TIEMPO DE ENFRIAMIENTO / *COOLING TIME* (MINUTOS)	≥ 40	
TEMPERATURA DE DESMOLDEO / *DEMOLDING TEMPERATURE* (ºC)	≤ 60	

Figure 5: Selected curing cycle.

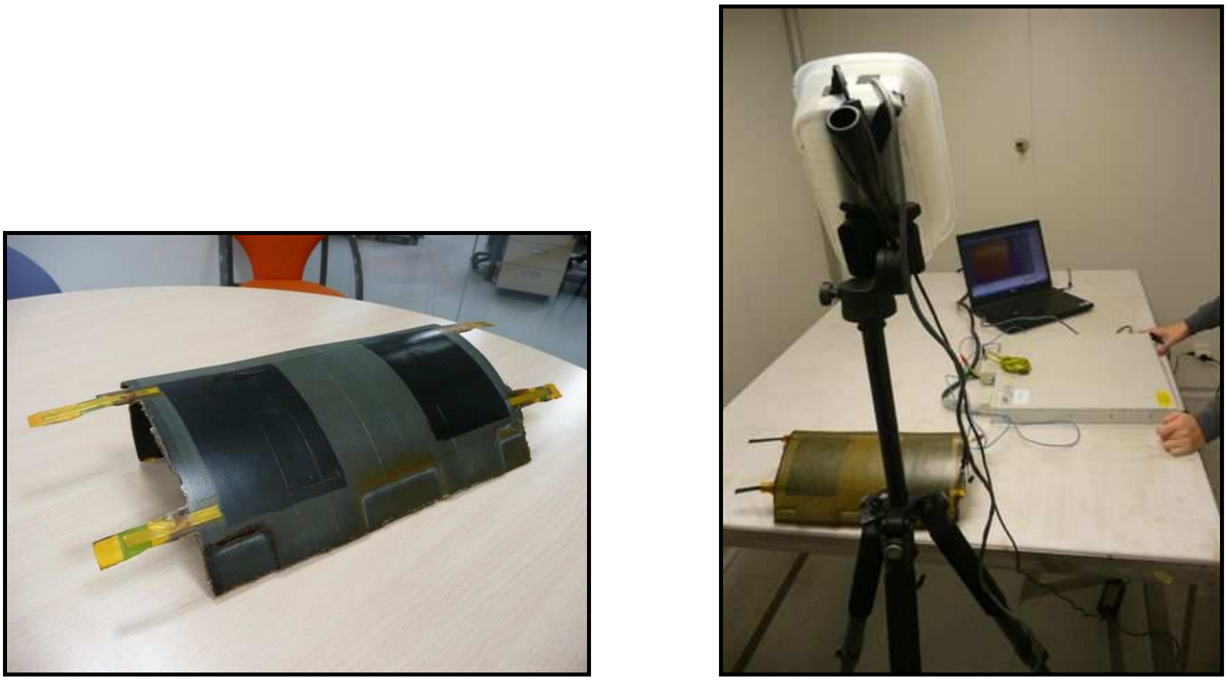


Figure 6: Demonstrator and Heating test on one of the demonstrators.

SIGNIFICANT REULTS
Testing of the functionalities shows that the concept developed in the project gives promising results.

- The heating performance seems to be adequate for an aircraft ice protection system in real service conditions. There is however some areas that could be important for future work: For example the integration of electrical connectors into the composite.
- When it comes to the fire properties the multi-layered composites have a higher pHRR (peak of heat release rate) than the reference system. The values are between 22-114 % higher depending on the multi-layered configuration. This shows that the addition of the multifunctional layer creates degraded fire behaviour in terms of pHHR. However, on the positive side the time to ignition is enhanced with some

configurations. To conclude, there is room for further studies in this area. However the importance of the fire behaviour is somewhat different depending on where the composite is placed in the application (for example inside the cabin or on the fuselage).

- FEM modelling analysis can give the "Effective Mechanical Material Model" of the nano-reinforced polymer material systems for both spherical and cylinder filler geometries. This is proved by the fact that the analytical values approach the experimental values. The work performed can hence be useful in the early stages of future product development.
- The tests on sensing capacity give some promising results. The developed method to integrate the fibres into the composite seems to be suitable. Also the pre-treatment of the fibres has been proved to be successful. Future work in this area should focus on enhancing the sensitivity of the sensing layer as well as develop methods to avoid disturbances on the signal due to for example temperature variations.

The main result of the project is to prove that he multifunctional concept is possible to manufacture within the framework of a traditional autoclave-prepreg process. Beside the fact that the processablity has been verified the tests performed and the demonstrators and prototypes also verifies the heating functionality and indicates that the concept has a successful sensing- and fire functionality.

WP4 Economic evaluation, exploitation and dissemination

This WP trends to cover the economic aspects of the developed technology, as well as exploitation of the innovation produced and dissemination plan. Required adjustment of conventional manufacturing processes has been an important aspect to evaluate economically. Steps required for certification of technology have been explored also.

In LAYSA project the evaluation of the results has been mainly focused in the concept probe of the developed multilayer composite. From the beginning of the project an initial configuration of the layer has been proposed but, during the development of LAYSA project, important changes have been accomplished, to assure the feasibility to manufacture a real part with the proposed multilayer concept.

A technical evaluation has been determined and the multilayer concept has been probed:

Development of Electrical/Thermal Conductivity

Raw carbon nanotubes are under the form of a light and disordered powder. For several applications more compact and ordered forms are needed. In particular ice protection devices that operate through Joule’s heating have ideally to be made of almost pure nanotube arrays or of composites with a large fraction of nanotubes.
Important work have done on the process parameters for the production of 1.5 and 2 wt.% CNT **masterbatches** based on epoxy resins. But direct loading of polymer matrices is a difficult approach for achieving large volume fractions of nanotubes. Viscosity increase and the presence of aggregates make a direct processing difficult. **Other approaches** are investigated in LAYSA with materials under the forms of **buckypapers** or **fibres.** Such forms are obtained from aqueous and homogeneous dispersions of nanotubes. These dispersions are then either membrane filtered or coagulated to obtain structures with a large fraction of nanotubes. Partners (Tec-INAS, UPPA-CANBIO and CRPP) contributions consisting in preparation and characterizations of these materials in order to selected the best option**. Regarding electrical/thermal conductivity, buckypaper approach reaches the objectives defined for this functionality.**

Development of Fire Resistance

Based on developments in WP1, it was decided to study the effect of nanoparticles on the fire resistance of composites without previous treatment. Innovative concepts based on gelcoats and alternative resins were proposed. Incorporation of nanoparticles in gelcoats has a **limited effect** on the reaction to fire. Use of **alternative resins** such as **benzoxazine** and **IEP22** is a **promising way**.
Processes for the synthesis of different composites were set up. Samples corresponding to various concepts were manufactured. **Nanomodified gelcoats**, **carbon or glass-fiber reinforced composites**, resins containing **nanoparticles** and **model resins** containing micrometric and/or nanometric fillers were investigated. The rheological and mechanical behaviour was also characterized for some of them. Due to difficulties in the resin manufacturing, a limited number of samples were produced.
The fire resistance of nanomodified gelcoats, resins containing nanoparticles and model resins containing micrometric and/or nanometric fillers was tested. Even if the behaviour of the final composite will be different from the one of the samples tested in WP2, due to expected interactions between the different layers, useful conclusions for the rest of the project can be drawn:

- For the development of a fire-resistant epoxy, sufficient **level of dispersion** of carbon nanotubes and montmorillonite in the resin is difficult to achieve.
- The **use of alternative resins** such as benzoxazine and IEP22 is valuable: the combination between good fire retardant properties and another functionality (electrical/thermal conductivity or monitoring) may be reached in the same layer.
- **Ammonium polyphosphate** and different types of **polyhedral oligomeric silsesquioxanes** provide good fire properties to the model resin. **The 50% pHRR decrease expected by the Consortium is reached with this system.**

Development of Sensing Performance

Several specimens, having different manufacturing history and material composition, were tested in INASCO and the conclusion we reached are stated below:

- Even though, some specimens had very low resistance, their sensing capability under strain 1% was measurable.
- In almost every test, specimen's resistance does not regain its initial value, but returns to a greater one after the end of loading. (hysteresis loop)
- Specimens with resistance in the order of kilo Ohm (10^3) exhibit greater maximum resistance values under the same strain percentage than the ones having a resistance in the order of Mega Ohm (10^6).
- Almost linear correlation between resistance and strain is observed in all tests.
- Finally, referring to samples that there were tested in both loading cycles, moisture adsorption might have caused small changes in resistance.

Modelling Analysis

Regarding modelling the main work carried out has consisted on the prediction of the electro-thermal behaviour, the mechanical response and the sensing performance of a nano-reinforced polymer material system taking into account factors that affect the relationship "structure (at nano-scale)–properties (at macro-scale)" by enforcing the multi-scale modelling principles. Moreover the modelling of the fire burnthrough response of a composite panel has been also considered.

Integration of the three functionalities in a prototype

When the most promising materials and processes had been identified, the optimization of the multifunctional concept and manufacturing process started. The work consisted of manufacturing trials and a subsequent assessment of the results. Identified problems were solved by making the necessary adjustments to the process before additional manufacturing trials were performed. When the optimization work was finished the manufacturing process was validated by the manufacturing of the demonstrators. A lot of work has been done on testing the demonstrators and prototypes according to a test plan and to assess the test results.

The main results are the following:

- The project has developed a multifunctional concept and integrated it into a traditional manufacturing process. The concept and manufacturing process has been validated.
- At the end, three demonstrators have been manufactured
- Guidelines for optimal performance
- Guidelines for future research necessary to increase the Technological Readiness Level
- Further investigations in electrode materials and electrodes integration into the composite part have to be accomplished.
- Further investigations in electrode materials and electrodes integration into the composite part have to be accomplished.

Economical Evaluation

From and economical point of view and related to the cost of the material, we can assure that in comparison with traditional materials used for electrothermal behaviour (copper mesh and copper sheets) the buckypapers solution is similar in cost.

It order to the buckypapers will be used at industrial level an automatic production way has to be set-up. One of the main objectives of ELECTRICAL project is oriented to achieve this goal which will allow the use of buckypapers at industrial scale.

In order to evaluate the manufacturing cost of the part, the mould and the curing cycles are the same that the used in a traditional composite part. For the production of parts an automatic buckypapers handling system has to be define, design and set-up to assure the economic feasibility of buckypapers.

4b) Exploitation Plan

The consortium of LAYSA project has been formed by 12 partners who have played different roles during the project and with different interests for the exploitation of LAYSA results.

a) END-USERS

AES

-Internal Difussion of Results
-Results Divulgation
-Synergy with AES Projects
-Further Developments
-Identification of Companies
-Start-up of components

b) RAW MATERIAL SUPPLIERS

CNRS-CRPP

-Supply of CNTs fibres.
-CNTs fibres industrialization

CANBIO:

-Supply of accurate nanoreinforced dispersions.
-Dispersions industrialization

ACG

-Production of nanoreinforced resins in bulk and film presentation
-Product industrialization

HUNSTSMAN

-Production of nanoreinforced resins and gelcoats.
-Product industrialization

c) TRANSFORMERS

TECNALIA

-Industrialization of Buckypaper Manufacturing Technology.
-Study of material combinations
-A Ph. D project
-Prototype manufacturing technology assessment.
-Internal Results Diffusion
-External Divulgation
-Identification of other Applications/Markets

SICOMP

-Analysis of proposed manufacturing technologies industrialization
-Internal Results Diffusion
-External Divulgation
-Identification of other Applications/Markets

INASCO

-Sensing capability measuring development.
-Internal Results Diffusion
-External Divulgation

ARICOM

-Company strategy

-Industrialization of prototype
-Further R &D-Project start-up of components -Technologies Industriallisation

d) RTD PERFORMERS

UP
-Electro/thermal, Fire resistance and sensing capability behaviour model developing.
-Publishing of the main results in international scientific journals.
-Presentation of the main results in scientific conferences.
-An improvement knowledge concerning the characterization methods for nano-scale modelling.
-A Ph. D project.

ENSCL
-An improvement knowledge concerning the fire resistance behaviour based on nanoreinforcements.
-Publishing of the main results in international scientific journals.
-Presentation of the main results in scientific conferences.
-A Ph. D project.

CU
-An improvement knowledge concerning the ice protection methods based on nanoreinforcements.
-Publishing of the main results in international scientific journals.
-Presentation of the main results in scientific conferences.
-A Ph. D. project

4c) Dissemination Activities

TECNALIA has been very active in the dissemination of LAYSA results. On one hand as Coordinator LAYSA project has been presented in different forums from March 2010 to December 2012.

-TECNALIA presented LAYSA project at EURONANOFORUM 2009 in Prague. INASMET-Tecnalia presented the LAYSA project at the EC organised EURONANOFORUM conference in Prague (2-5 June 2009). In Prague the LAYSA presentation was given to a large audience of researchers and EC officials creating hot interest for the applications of nanotechnology in the Aeronautics and generally in the Transport sector.

-TECNALIA presented LAYSA project at 6th ECNP, Conference on Nanostructured Polymers and Nanocomposites, 28-30 April 2010, Madrid. Poster Contribution**:** “Multifunctional Layers for Safer Aircraft Composites Structures.”

-HUNTSMAN will present some results of LAYSA project at ECCM 14 Conference in Budapest, Hungary, 7-10 June 2010. ”Refining the thermal and electrical properties of aerospace epoxy resins”

-C. Elizetxea, M. Txapartegi, A. Iriarte.
Multifunctional layers for safer aircraft composite structures
“AERODAYS 2011”, Sixth European Aeronautics Days, Madrid (Spain), 30th March-1st April 2011.

-A. Iriarte, M. Chapartegui, C. Elizetxea.
Multifunctional Layers for Safer Aircraft Composite Structures
“MATCOMP 2011”, Girona (Spain), 5-8th July2011.

After the end of LAYSA projects it has been also presented in a important International forum, JEC Composite Show 2012 with the agreement of all the involved partners.

-TECNALIA Booth X23. ***“JEC Composite Show 2012”.*** 27-29th March 2012.
In figure 65 it is showed the brochure of LAYSA project distributed among the people who presents interest about the project in last JEC show 2012,

Figure 7: LAYSA project brochure for JEC Composite Show 2012.

TECNALIA as Technical partner of LAYSA project, has been involved in the development of a new electrothermal heating system based on CNTs buckypapers. As a result of the research carried out in LAYSA project, Maialen Chapartegui has passed her PhD examination in December 2012 and he has contributed in the dissemination of LAYSA project as a result of the five publications accepted in top ranked European journals:

- **M. Chapartegui**, N. Markaide, S. Florez, C. Elizetxea, M. Fernandez, A. Santamaría, "*Specific rheological and electrical features of carbon nanotube dispersions in an epoxy matrix*". Composites Science and Technology, 70 (2010), p. 879–884.

- **M. Chapartegui**, N. Markaide, S. Florez, C. Elizetxea, M. Fernandez, A. Santamaría, "*Curing of Epoxy/Carbon Nanotubes Physical Networks*". Polymer Engineering & Science 2012.

- **M. Chapartegui**, S. Florez, C. Elizetxea, M. Fernandez, A. Santamaría A., "*Carbon nanotubes accelerate epoxy resin curing*". Society of Plastics Engineers–Plastics Research Online. 10.1002/spepro.003891

- **M. Chapartegui**, J. Bárcena, X. Irastorza, C. Elizetxea, M. Fernandez, A. Santamaría., "*Analysis of the conditions to manufacture a MWCNT buckypaper/benzoxazine nanocomposite*". Composites Science and Technology, 72 (2012), p. 489–497.

- **M. Chapartegui**, J. Barcena, X. Irastorza, C. Elizetxea, E. Fiamegkou, V. Kostopoulos, A. Santamaría., "*Manufacturing, characterization and thermal conductivity of epoxy and benzoxazine MWCNT buckypaper composites*". Accepted in May 2012, for Journal of Composite Materials publication.

ENSCL is involved in WP4 for the dissemination of the results. Caroline Gérard was hired by ENSCL to work for Laysa in the framework of PhD project (her salary was paid by Laysa for 3 years). She passed her PhD examination in June 2011 and since her dissertation is not confidential, it is now downloadable from the website of the library of the University of Lille. It contributes then to the dissemination of the Laysa project and it proves the scientific quality of the results obtained in Laysa.

In addition to this, her work has been partialy published in top-ranked journals (other papers are in preparation):

-S. Bourbigot, G. Fontaine, A. Gallos, C. Gérard and S. Bellayer,
FUNCTIONALIZED-CARBON MULTIWALL NANOTUBE AS FLAME RETARDANT FOR POLYLACTIDE,
Fire and Polymers, ACS Symposium Series 1013, Editeurs: Wilkie, C.A. et Nelson, G.L, Pub. ACS (2009), 25-34

-C. Gérard, G. Fontaine et S. Bourbigot
NEW TRENDS IN REACTION AND RESISTANCE TO FIRE OF FIRE-RETARDED EPOXIES
***Materials,* 3, pp. 4476-4499, 2010**

-C. Gérard, G. Fontaine and S. Bourbigot
SYNERGISTIC AND ANTAGONISTIC EFFECTS IN FLAME RETARDANCY OF AN INTUMESCENT EPOXY RESIN
***Polymer for Advanced Technologies,* 22(7), pp. 1085-1090 2011**

Laysa results have been also presented at international conferences (oral presentations):

-G. Fontaine, C. Gérard, S. Bellayer et S. Bourbigot,
FIRE RETARDED EPOXY RESIN: IMPRESSIVE SYNERGISTIC EFFECT OF APP/POSS COMBINATION
«2010' International Symposium on Flame-Retardant Materials and Technologies - ISFRMT 2010 », Chengdu (China), September 2010
-G. Fontaine, C. Gérard, S. Bellayer et S. Bourbigot
POSS A SYNERGIST FOR EPOXY RESIN (FIRE RETARDED WITH APP): MECANISM OF ACTION
«22th BCC Conference - Recent Advances in Flame Retardancy of Polymeric Materials», Vol. 22, 10 pages, Stamford, CT (USA), May 2011

C. Gérard, G. Fontaine et S. Bourbigot
CARBON NANOTUBES: FLAME RETARDANTS FOR EPOXY RESINS.
«Eurofillers 2011», 6 pages, Dresden (Germany), August 2011

4. POTENTIAL IMPACT

The trend in aerospace sector is to use composites for more components in civil and military aircraft and spacecraft. Around 9% of the total composite shipments in Europe (1.540.000 tonnes and 5,2 BEuro) are used in aerospace / defence. **The current trend is to reduce weight, so lightweight composite materials have been increasingly used**.
However, concentrating solely on structural mass reduction does not lead to further lowering of equipment mass because the structure typically represents as little as 10 - 15% of the total mass.
The envisaged solution is to design structural elements that can integrate multiple functions, known as multifunctional structures. The development of nancomposites predicts an opportunity to fabricate components with multifunctionality.

Breakthroughs in nanomaterials science, production and functionalization are leading to developing new applications.

In the conventional aircraft, ice protection functions, fire protection functions and health monitoring of composite structures are designed and fabricated into separate elements; the socio-economic strategic impact of nanomaterials technologies that will be investigated in LAYSA project by the **integration of those three functions (ice/fire protection and health monitoring) in an unique system** has one main aspect for aerospace; nanomaterials technologies open the door to high performance, environment-friendly and safer aircraft operation by better exploiting available multifunctionality potentials derived from their exceptional properties, in terms of thermal and electrical conductivity and sensing capacity.

On one hand, LAYSA outputs are expected to have essential societal impact due to saves in manufacturing process and fuel consumption as a consequence of the replacement of three systems by a unique lighter multifunctional system.

On the other hand, the technologies and tools that will result from this proposal are essential to increase the European market from the current level in the next 10 years, and they will offer opportunities for the employment of highly skilled professionals. This would contribute in solving of heavy societal problems interconnected with the high unemployment in Europe.

Summarizing, a successful LAYSA project will ensure a strong strategic impact and will have clear Socio-Economic benefits within the next five to ten years by contributing to:

- Enhance European aeronautic industry competitiveness
- Enhance European employment
- Meet societal needs for more environmental friendly, safer and efficient air transport.
- Meet societal needs for more environmental friendly, safer and efficient manufacturing

- **<u>Innovation related activities</u>**

The limitations of composite structures for thermal, electrical and fire performance have been a drawback to extend its use in many applications. LAYSA project aims to develop a novel nanocomposite multifunctional layer to be integrated into composite structures with combined properties in terms of electrical / thermal conductivity, fire protection and sensing capabilities. This will be developed by exploiting the capabilities offered by Nanomaterials MMs, specially Carbon Nanotubes CNTs and Carbon Nanofibres CNFs. Low volume additions of those materials (1-5%) provide property enhancements with respect to the neat resin that are comparable to that achieved by conventional loadings of traditional fillers (15-40%), even unique value-added properties not normally possible with traditional fillers are also observed, such as reduced permeability, tailored biodegradability, optical clarity, self-passivation and flammability, oxidation and ablation resistance. In addition, the lower loading facilitate processing and reduce component weight increase.

The expansion of these unique opportunities requires the development of some relevant aspects such as suitable dispersion and orientation of NMs in the matrix and a strong bonding between them, incorporation and alignment of such structured NM assemblies into composite materials, and design of those materials taking into account the integration of different functionalities.

Although several final applications are predicted for nanocomposites and different groups are working on them, currently there are no composite products in the market with nanocomposite technology Incorporated.

The main innovations of LAYSA project reside in:

- A nanocomposite layer with proper electrical and thermal conductivity for ice protection, improved fire behaviour and intrinsic sensing capabilities for health monitoring.
- Development of modeling tools to analyze and design novel multifunctional layers based on Nanomaterials
- Integration of multifunctional layer in composite structures.

Current methods	Technical limitations and issues	LAYSA improvement
Ice protection Electrothermal system, where heat is applied by flexible pads adhesively bonded on the surface of composite structure, or molding heating element (foil, film, resistance wire or mesh) into the surface of the composite structure during manufacturing process.	• Additional temperature sensors are required. • They add laborious operations and manufacturing complexity during the component manufacturing. • They require intense inspection and maintenance to make sure that they will function properly during precarious phases. • Important weight increase (1-2 kg/m2), electric power consumption (11-49 kW/m2) and aerodynamic performance penalty. For that, it is not possible to use for all aircraft composite structures. • Element burnout is of concern since no regulation exist and failure is total.	• **Temperature sensors are not required**, because of self-sensing capability of the layer. • Important saves due to **simplification of manufacturing processes and maintenance operations** • **No aerodynamic performance penalties** • Important **weight reduction** due to elimination of heavy heating elements. Possibility of heating all aircraft composite structures • Element bournout is not of concern because of improvement of fire resistance • **Reduction in power consumption** due to unsurpassable current density of considered nanomaterials
Fire protection • Use of additional thermal barrier coatings on the structures. • Chemical reformulation of matrix. • Addition of additives and fillers into the matrix.	• The barrier coatings must be periodically inspected, repaired and maintained in very good conditions in order to maintain their effectiveness. • The fillers have in many cases negative influence on the processability and mechanical properties of the resin itself. • Chemical reformulation of the resin only achieves a partial improvement of fire properties. Combination with other method is required.	• With respect to all methods, **improved fire behaviour**. • With respect to barrier coatings, important saves due to simplification of manufacturing processes and maintenance operations • With respect to fillers, easiness of manufacturing process and weight reduction
Health monitoring Fiber optic or piezoelectric sensors embedded into the composite structure.	• Fiber optic diameter in many cases acts as stress concentration site, gives low strength at fiber-splicing locations, and they need for electro-optical signal conversion modules • Piezoelectric sensors have low induced strain capability, high density, brittleness and limited fatigue life.	• The new integrated system does not have any detrimental effect on the structure and does not suffer by fatigue related problems. • It will be possible a continuous health and usage monitoring and self-inspection; summarizing, a **"smart maintenance"** will be achieved.

- **Added value in carrying out the project at European level**

The proposed technology implies a multi-disciplinary approach, within the collaboration of electrical engineers, icing and sensing technologies experts, fundamental researchers in nanocomposite technologies, as well as composite structure designers and manufacturers. Addressing the technology needs of future aircrafts, new design approach has been proposed with LAYSA for improvement composite structures safety and security in terms of ice and fire protection, as well as health monitoring. Therefore, the frame of an STREP project within a multidisciplinary integrated team is perfect to gather all the skills needed to validate new concept. That competent integrated team offers for the LAYSA the key advantage to cover all technical fields involved in such a technology with materials experts, engineering experts, design experts, manufacturing experts, testing capabilities, promoting and diffusion services.

The effort needed to meet the techno-economic targets of LAYSA could not be carried out on a national level because:

- Specific knowledge on electrical-thermal-fire- sensing behaviour of nanocomposite materials, as well as icing technologies is dispersed in different European centres involved in R& D activities (TEC-INAS, SICOMP, CNRS-CRPP, University of Patras, University of Lille, University of Cranfield, University of Pau-CANBIO) located in several European countries.
- Sectors selected reflect also main industrial actors in the various countries as HUNTSMAN and Advanced Composite Group for raw material manufacturers, INASCO as sensors technology supplier, ARIES COMPLEX as aircraft part manufacturer and AERNNOVA as end-user.

The multinational approach has two clear advantages:

- Provide the critical mass of researchers –experts on the technological fields needed for LAYSA
- The joint added value of the partners together is higher that the added value of each individual partner.

- **<u>Individual Exploitation and Dissemination Strategy</u>**

Each partner has their own specific strategy to exploit and disseminate the results of the project:

TECNALIA

It is anticipated that the knowledge gained in LAYSA will strengthen our position as a niche technology provider in the nano-materials area and especially in the advanced and modern processing techniques. In addition, the expertise gained will allow us to widen our offer to other markets and other customers. TEC-INAS is interested in watching out for possible patents or IPR to try to exploit them through our incubator NAHISA or to transfer the technology developed under LAYSA on new sectors such as railway or windmill blades. An exploitation plan is envisaged along the project.

SICOMP

The focus for SICOMP will be on exploiting the knowledge from this project into profitable products for their customers. This project will strengthen scientific knowledge and development capabilities on nano-technology and functionalized materials and lead to increased sale of consultancy services on processing and design of functionalized composites and nanotechnology in general. An estimated value of these consultancy services is 400 k€ during a two-year period. Based on previous experience, their consultancy services will increase their customers' sales approximately 10 times their sale, i.e. 4 M€.
SICOMP plans to market the acquired competence to existing customers in the industrial sectors of aerospace, automotive and electrotechnical industry. New customers and industrial sectors will be identified during the duration of the project.

CNRS-CRPP

CNRS-CRPP plans to disseminate the results of the project for the exploitation of carbon nanotube microfibres in aerospace applications. This way, it is expected that CNRS-CRPP will be able to provide a European industrial turnkey solution based on the use of carbon nanotubes in smart composites within 3 years from the project end. Collaboration with LAYSA partners is envisaged to be essential for the success of CNRS-CRPP´s exploitation plan.

UNIVERSITY OF PATRAS

UP's plans for dissemination and exploitation are as follows:

1) Presentations and publications at dedicated seminars, conferences and workshops
2) Publications in recognised national and international journals.
3) Collaboration with SMEs involved in the monitoring business as well as with larger industries and public authorities looking at the application of developed tools in their structures and systems.

UNIVERSITY OF LILLE

It is expected that LAYSA project will reinforce our expertise in the field of multifunctional nanocomposite-based materials. LAYSA project should also permit to develop new collaborations between laboratories and industrial partners to share our knowledge and experience. UOLI will disseminate the information concerning the LAYSA activities via lecturing, seminars and contacts with local media, promote local developments on a broad scale on conferences and workshops, and writing scientific notes and design guidance, and publications, and play a visible role in the global development of the LAYSA participation in appropriate working groups.

UNIVERSITY OF CRANFIELD

We will use this opportunity to train a student in this growing and highly interdisiplinary area. The project will bring thgether workers in two schools and reinforce existing links with the Cavendish Laboratories (Cambridge University) assembling a wide range of capability and knowledge. We will be able to publish some of the work and expect to use the knowledge and momentum gained to work on further developments in the application of nanocomposites to a broad range of applications.

INASCO

INASCO's expertise in monitoring and control systems ranges from the material-state based control in composites processing to the prognostic and diagnostic technologies for structural health monitoring. It is anticipated that the technologies of CNT sensors/actuators to be developed within LAYSA will complement and enhance the commercial sensor technology as well as their monitoring and control systems and apply them to more complex parts and in various sectors. Scientific publications and presentations are also envisaged once the technology is safeguarded through patents and IPR's. In a longer term, the implementation of LAYSA technology will significant boost the sales of the currently available dielectric monitoring system in the aerospace sector by expanding the application range and the versatility of the sensing. The direct turnover increase could be in the area of 500 kEuro over a two-year period after the implementation

UNIVERSITY OF PAU-CANBIO

It is expected that LAYSA project will reinforce our expertise in the field of multifunctional nanocomposite-based materials, especially those based on carbon nanotubes. LAYSA project should also permit the development of new collaborations between laboratories and industrial partners to share our knowledge, experience and tools. Canbio will disseminate the information concerning the LAYSA activities via lecturing, seminars and contacts with local media, promote local developments on a broad scale on conferences and workshops, and writing scientific notes and design guidance, and publications, and play a visible role in the global development of the LAYSA participation in appropriate working groups.

ADVANCED COMPOSITE GROUP

ACG has built its business on developing unique and innovative resin systems which will be leveraged in this project to solve processing difficulties and to provide a high-performance, cost-effective polymer matrix solution for the aerospace market application. Following from this development, exploitation of the technology is anticipated from direct sales within the aerospace community and from transfer of the technology to other markets including: construction, automotive and energy infrastructure.

HUNTSMAN

Since years and with major success, HUNTSMAN provides Composite world with building blocks (individual components –resins, hardeners, toughners…etc - for formulators) and/or formulations (adhesives, structural matrices for prepregs, direct process …etc).

Expertise gained participating to this Aerospace project will allow HUNTSMAN to promote "nanomaterials modified specialty epoxy resins" technology in several other major markets for which requirements are more and more close to Aerospace ones : Windmill, Marine, Automotive …

As an epoxy resin manufacturer, HUNTSMAN aims to develop the use of epoxy resins and hope to increase market shares proposing on the market unique specialty products based on the nanomaterial technology developed during this Laysa program.

ARIES COMPLEX

The idea with respect to the LAYSA project is to acquire the knowledge in the field of multifunctional nanocomposite-based materials, especially those based on carbon nanotubes in order to know their characteristics to be able to make structural designs with these new materials and to apply these kinds of designs to our final products. This project will also permit the establishment of new collaborations between Universities, Technological, Centers and our company.

AERNNOVA

The LAYSA project targets contribute to the strategies of AERNNOVA whose strategy is focused on large and complex structures by providing the right tool to submit winning proposals.

- **LAYSA Project Results**

No.	Self-descriptive title of the result	Category*	Partner(s) owning the result(s) (referring in particular to specific patents, copyrights, etc.) & involved in their further use	
			OWNER	USER
1	Production of CNTs fibres	B	CNRS-CRPP	ALL
2	Production of accurate dispersions of nanoreinforcements	B	CANBIO	ALL
3	Production of nanoreinforced resin films	B	ACG	ALL
4	Production of nanoreinforced gelcoats	B	HUNTSMAN	ALL
5	Production of Buckypapers	B	TECNALIA	ALL
6	Sensing capability measuring protocol	B	INASCO	ALL
7	Models for electrothermal, fire resistance, sensing and multifunctional behaviour.	B	UP	ALL
8	Setting-up of processing technologies: -Resin Transfer Technology (RTM) -Autoclave	B	SICOMP ARICOM, TECNALIA	ALL
9	Guidelines for multilayer composite parts manufacturing	B	AES, ARICOM, SICOMP and TECNALIA	ALL

* A: results usable outside the consortium / B: results usable within the consortium / C: non usable results

5. THE ADDRESS OF THE PROJECT PUBLIC WEBSITE

Creation and maintenance of the project website:

www.laysa.eu

which includes the updated information of the project.

The website is divided in two different areas, one for public dissemination of the project and, the second one, only for project members.

Figure 2: Website of the LAYSA project.

ANNEX A: Project Member Contact List

ACP7-GA-2008-213267

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