EUROPEAN SPACE AGENCY
CONTRACT REPORT

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1 Introduction

1.1 Scope of the document

The scope of this document is to provide an overview of all the work done under NESTOR activity. The document briefly covers the whole activity, from the investigation of suitable strategies for nano-enabled fibre reinforced polymers and proof of concept provision, to the preliminary material development and definition of the “Made to Measure” concept. A brief presentation of the company’s quality system related to neFRP production is also included.

1.2 Applicable Documents

[AD1] Statement of Work – Development of Nano-Enabled Fibre Reinforced Plastics to RfQ 3-13747/12/NL/AF

[AD2] Technical Proposal - Study on strategies towards the use Nano-Enabled Fibre Reinforced Plastics in Space to RfQ 3-13747/12/NL/AF, December 2012

1.3 Reference Documents

[RD1] ESA-NESTOR-TN1-UOP: State of the Art Review on ne-FRP materials and processes

[RD2] NESTOR-TN2-ADCO: Space industry requirements for FRPs

[RD3] NESTOR-TN3-ADCO: Trade-off criteria, weighting factors, and methodologies for selection of nano-enabled applications

[RD4] NESTOR-TP1-UOP: Preparation of test plan for nano-enabled composites

[RD5] NESTOR-TR1-UOP: Test report for nano-enabled composite processed

[RD6] NESTOR-TN4-ADCO: Benefit analysis for the utilisation of neFRP in space systems

[RD7] NESTOR-TM5-ADCO:Quality assurance for neFRP: System design and documentation structure including manufacturing procedures

[RD8] NESTOR-TN5-ADCO: Survey and design of quality assurance system for nano-enabled FRP

[RD9] NESTOR-TN7100-ADCO: Systematic approach formalisation process for nano-enabled FRPs

1.4 Abbreviations

AD	Applicable Document	neFRP	Nano-Enabled FRP
CDR	Critical Design Review	PDR	Preliminary Design Review
DD&T	Design, Development and Test	PM	Progress Meeting
ESA	European Space Agency	QA	Quality Assurance
FP	Final Presentation	RD	Reference Document
FR	Final Review	RTM	Resin Transfer Moulding
ICD	Interface Control Document	S/C	Spacecraft
ID	Interface Document	SOW	Statement Of Work
IRD	Interface Requirement Drawing	TN	Technical Note
LRI	Liquid Resin Infusion	TRR	Test Readiness Review
MTR	Mid- Term Review	TBC	To Be Confirmed
N/A	Not Applicable	TBD	To Be Defined
CNT	Carbon nanotubes	WP	Work Package
FRP	Fibre Reinforced Plastics		

2 Scope and overview of the activity

The scope of the project was to investigate and develop suitable strategies for the development of nano-enabled fibre reinforced plastics (neFRP) in order to produce materials tailored to the customer's needs and requirements. Within the scope of the work was also to establish a quality control system to assure that the produced products are of acceptable quality and can be integrated in functional parts for space applications.

The work started with a literature review of suitable nano-enabling techniques for two manufacturing processes: autoclave and infusion-based/ RTM composite manufacturing. In parallel, a market survey was performed to identify the potential space applications where the use of such novel materials would bring benefits. Based on these, a trade-off of the techniques was performed and certain routes were identified for the company to proceed on. A computational benefit analysis validated the concept and proved the applicability of ne-FRPs in space applications.

A carefully planned material development and testing was performed in order to validate the concept in a real-life environment. The results of these tests are used as an initial database of materials in order to describe the final and most crucial step, the "Made to Measure" process. "Made to Measure" materials are tailored to fit a specific customer's needs and are the main concept around which the project is revolved. This concept and the steps leading to it are also defined under the framework of this project.

In parallel, a quality system was established from a managerial and a production point of view. The company's management system was classified in sub-categories and the exact steps taken for the implementation of each stage are described in details, in order to reassure that the procedure is stable. With respect to the production's quality assurance, the critical parameters for each intermediate product during the course of the process are identified and suitable techniques to monitor each of them are investigated. At the end of the process, a detailed quality assurance plan to be implemented was decided.

3 Market and literature survey for ne-FRP technology

3.1 Nano-enabling techniques identification

The first step was to perform a literature review on the already available techniques for neFRPs. The review was performed with the composite manufacturing methods in perspective and taking into consideration the feasibility of near term application of the technologies. The main focus is placed on the two most commonly used production methods and their related technologies; Autoclave and RTM. With these principle methods covered, it is believed that the majority of composite production technologies are addressed and that the rest can benefit from similar approaches.

For each of the manufacturing methods, the end-to-end process is identified and mapped. To deliver a final neFRP product, a change in the conventional processing steps is required. For each identified step, potential nano-enabling concepts to intervene to the process and integrate the nano-phase in the FRP, thus delivering a neFRP product, are addressed.

In principle, the main steps to introduce the nano-phase into the composite are two; either the raw material should already contain the nano-phase, or the nano-phase can be integrated during the lamination process. An additional step in the chain may be introduced between the supplier of the raw material and the manufacturer, where the nano-phase can be integrated to the material. For the RTM process, as the two major constituents of the composite (fibres and matrix) are assembled in different steps, the nano-phase can additionally be introduced during the injection of the resin.

Following, the techniques identified for the integration of the nano-phase during composite neFRP production via Autoclave and RTM are presented.

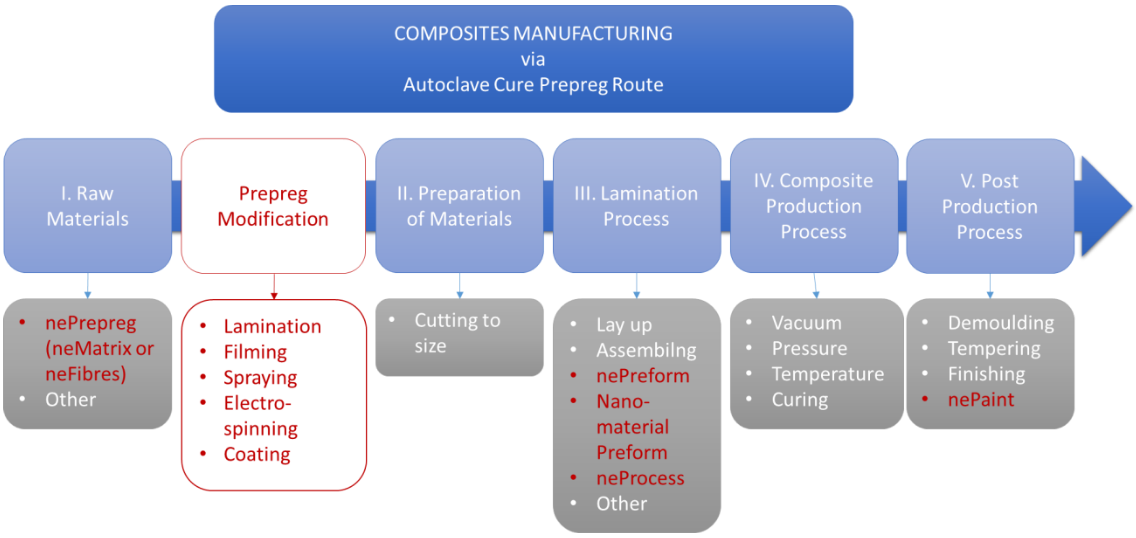


Figure 1. Autoclave Composite Manufacturing Process: Including Nano-Enabling Steps (indicated in RED)

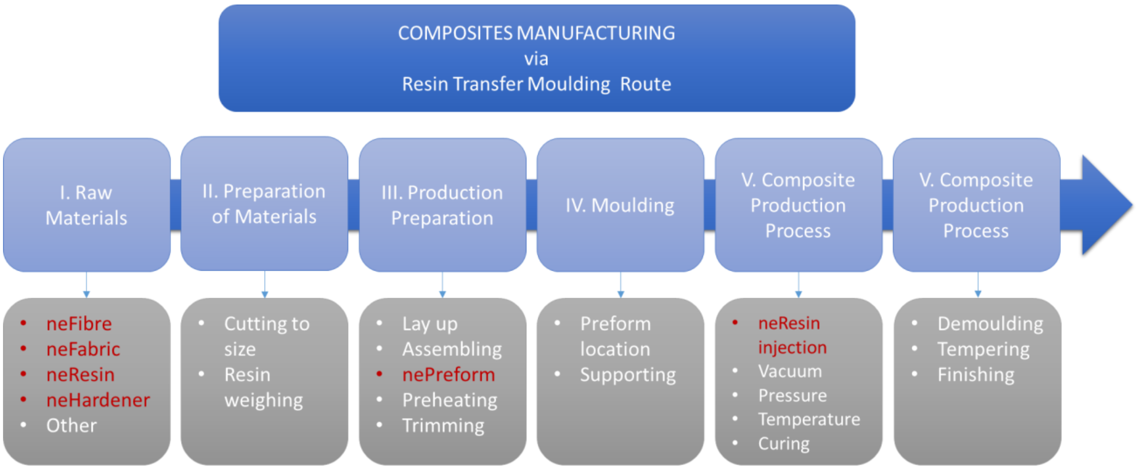


Figure 2. Infusion-based Composite Manufacturing Process Steps: Including nano-enabling steps (indicated in RED)

3.2 Market survey and space applications identification

Subsequently, in order to have a better understanding of the needs in the space industry, a survey to potential customers was conducted and potential applications for the technologies developed were identified. Through direct contacts with end-users, the existing requirements as well as the improvements sought after through the implementation of nano-integration technologies were established.

Having established a list of potential customers for the nano-enabled material, the next step was to select a limited number of them for the extension of the survey. The selection was based on certain criteria, with the most important being the suitability of ADAMANT's product(s) for the needs of the customer's project. From the products identified during the market survey, the ones that cover these criteria are **cubesat/solar panels** and **radiators**.

3.3 Trade-off analysis of nano-enabling techniques

A trade-off tool was developed, which combines the above parallel investigations in order to help in the selection of the most appropriate ne-FRP applications for space.

The trade-off was divided into two main parts in order to help evaluate the nano-enabling technologies through more specified criteria; The *industrialization analysis* was based on the criteria connected with the adoption of these technologies into manufacturing, and the *potential applications suitability criteria analysis*, based on criteria connected with the material properties. At the end of the rating process, industrialization scoring is summed with potential application scoring in order to provide final scoring per application.

For each nano-enabling technology a final rating is derived as a sum of the technique's rating for industrialization criteria and potential applications criteria, weighted with appropriate factors. Based on the described trade-off tool, the final selection of the more promising nano reinforcing routes per application was obvious and can be summarized below:

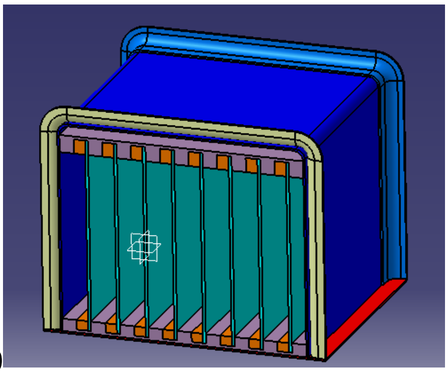
- Integrating a *nano-doped resin film (FILM)* into the composite is a method that is expected to satisfy all space applications and industrialization criteria and is applicable both to Autoclave and RTM.
- For Autoclave manufacturing, integrating nano-phase on prepregs (nePrepreg) is also promising and can be achieved in three ways: by *powder scattering-coating*, by inserting nano-phase with the *lamination of nano-doped film* or by depositing a *nano-doped slurry*.
- For RTM manufacturing, *inserting the nano phase into the matrix* or *veil technology* seems to satisfy the criteria set and can be considered good alternatives to *nano-doped resin film*.

➢ **It must be noted that under the framework of NESTOR, Autoclave composite production and powder scattering was the area of greater focus.**

4 Benefit analysis of ne-FRPs

Typically, S/C have several electronics housings, made of aluminum. The tendency to reduce S/C mass and subsequently the cost of launching has led to the increased use of composites in spacecraft structures. The advantage of composites for housings comes from their superior stiffness and strength characteristics compared to aluminum. However, CFRP housings have inferior electrical, thermal and particle radiation properties than aluminum.

A study was performed to investigate the possibility of utilizing nano-enabled composite materials for electronics enclosure. The prepregs selected for this study are the ones most commonly used for S/C structures and electronics housings. The thermal conductivity properties of nePrepregs were scaled up according to predictions of their achievable performance enhancement.

a) 

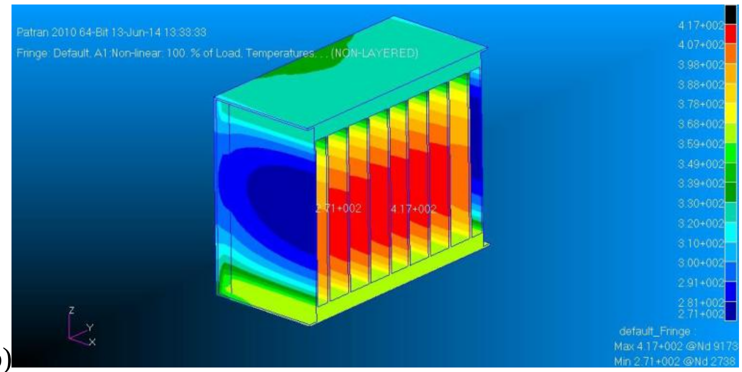
b) 

Figure 3. a) CAD model of the housing, PCBs in place and front face removed, b) Temperatures for 100% of the maximum power input for nano-enabled composite electronics housing

The main outcome of this study was that if the nano enhancing of the thermal conductivity is possible at the extent that it was assumed, an electronics housing made of a neFRP can be comparable in terms of thermal performance and mass with an aluminum one. A specific prepreg was distinguished as a possible candidate for further development and testing.

5 Material developments and testing

A preliminary material development and testing campaign was conducted as a next step in order to assure the feasibility of the selected techniques experimentally and validate the neFRPs concept.

5.1 Test plan

The developments of neFRP under NESTOR project are targeting specific directions seeking improvements through the use of nano-reinforcing concepts. These directions are identified as Material Development Streams (MDS). An overall assessment of all materials was out of the scope of this campaign. Tests directly assessing the targeted performance by each stream have been performed. An overview of the identified material development streams together with the respective targets is provided in Figure 4.

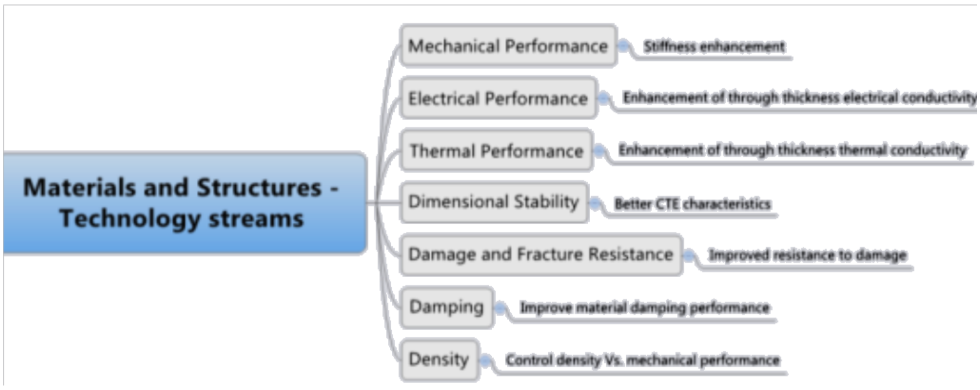


Figure 4. Material development streams

For the MDSs prioritization, the streams are assessed under two perspectives:

- How important is currently the property they cover for space applications? This includes whether the performance has been achieved by other means and how much interest the property has attracted the past years.
- How confident are we that the performance is going to be achieved? Has it been achieved by others on material level by some means?

The next step in the process was the identification of relevance and matching of the neFRP technologies and MDSs, as seen in Table 1. This table is populated for the four dominant streams. The assessment was done on whether the process is applicable or not for achieving the targeted performance. If yes, then it was assessed whether the process knowledge and experience is available or can be developed by Adamant Composites or University of Patras within NESTOR project.

A plethora of nano-/micro-sized materials were identified, assessed and documented for the targeted properties. In total 15 neFRP concepts-plates were finally produced. More than 100 specimens were prepared and tested.

Finally, the manufacturing plan was established, based on a combination of different feasible nano-enabling processes and performance design drivers. Composite plates reinforced with different materials and areal weights have been manufactured and tested.

All of the produced materials were subject to a quality control comprising visual inspection, fibre volume fraction estimation and ultrasonic testing.

Table 1. Matching of neFRP processing technologies with MDS

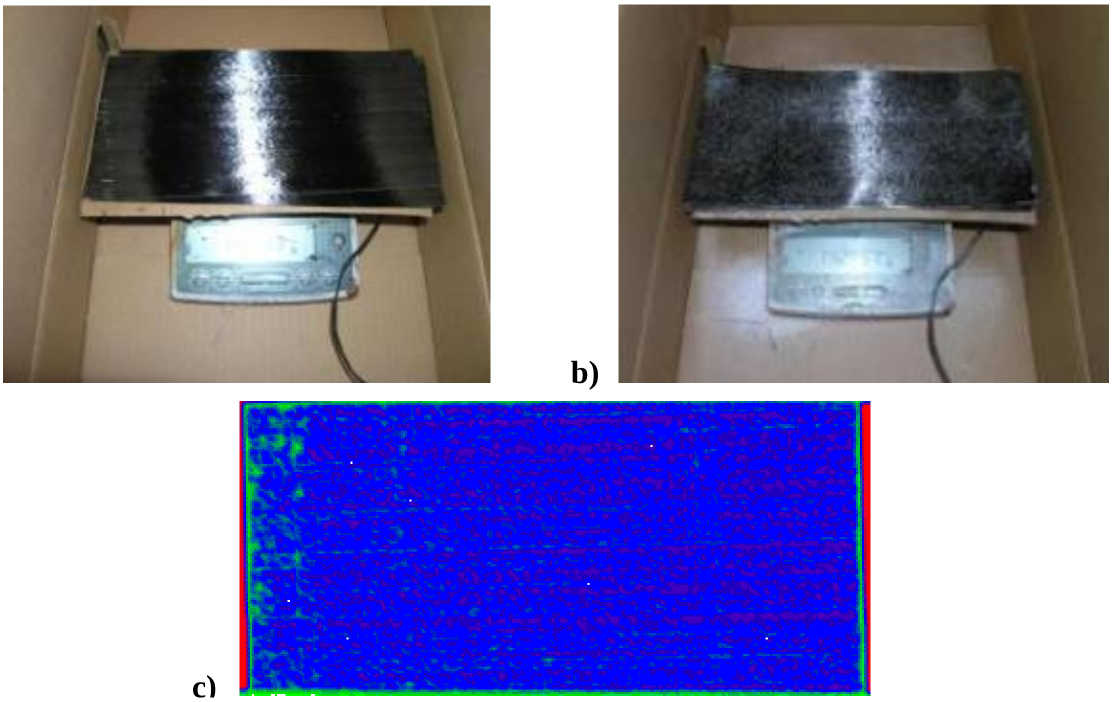
Potential additives:	Process 1	Process 2	Process 3	Process 4	Process 5	Process 6	Process 7	Process 8	Process 9	Process 10						
Development stream											Batch 1	Batch 2	Batch 3	Batch 4	Batch 5	Total
Thermal performance	-	O	-	O	-	11	-	O	-	-	-	2	5	-	4	**11**
Damping	O	1	O	O	-	2	O	-	-	-	-	1	-	2	-	**3**
Density	-	-	-	O	-	1	-	O	-	-	1	-	-	-	-	**1**
Electrical performance	O	-	-	O	-	-	-	O	-	-	-	-	-	-	-	**0**
Mechanical performance																
Dimensional stability																
Damage and Fracture resistance																
																15

** - : not applicable*

*** O : applicable but not achievable within NESTOR*

5.2 Test results

In general, the majority of the plates exhibited good or acceptable quality, while the fibre volume fraction of the plates did not seem to be severely degraded by the increase of the areal weight of the nano-enabling agent.



a) b) c)

Figure 5. a) Conventional prepreg, b) Treated prepreg, c) Ultrasonic C-scan image showing good consolidation and high quality of the produced plate

Comparative graphs of the results from all the manufactured scenarios are exhibited below.

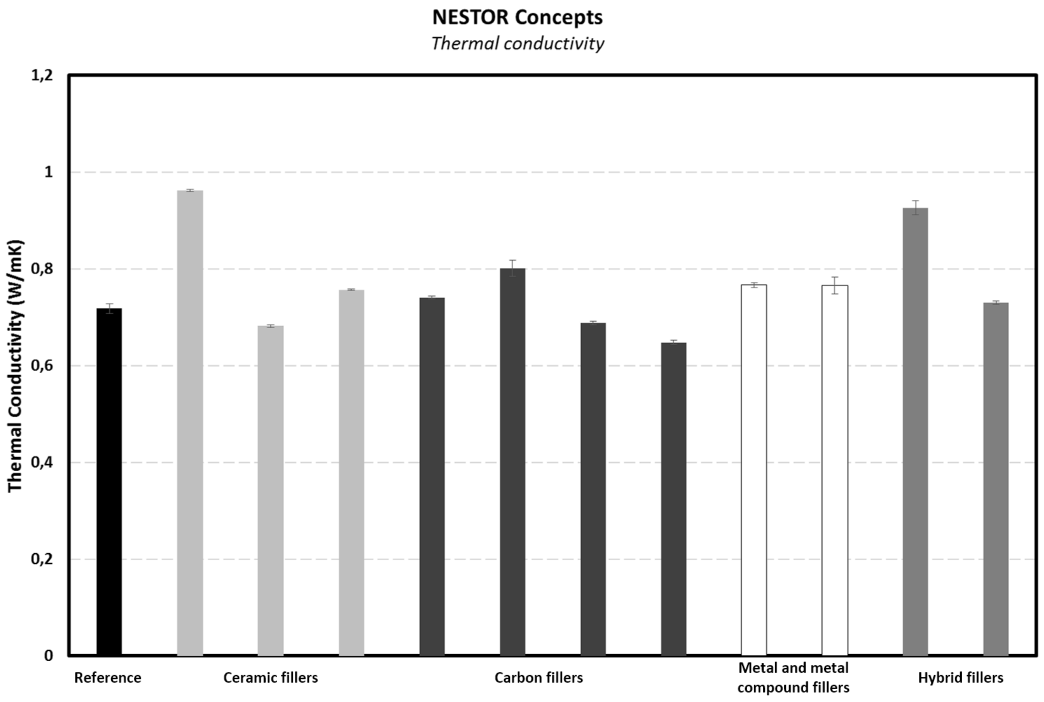


Figure 6. Thermal conductivity of all concepts developed under Thermal MDS

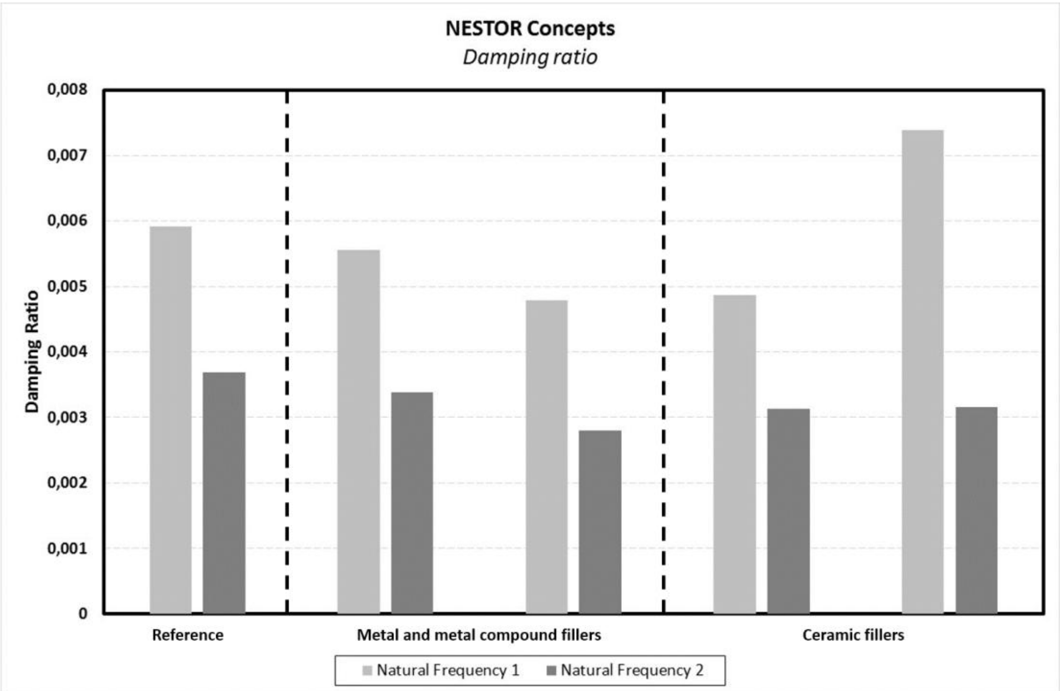


Figure 7. Damping ratio of all concepts developed under Damping MDS

6 Quality assurance for neFRPs and management system

6.1 System design and documentation structure

Adamant Composites LTD has tried to design a QS in accordance with the requirements of AS 9100, ISO, ECSS and other standards. The system will be continually improved through the use of the quality policy, quality objectives, audit results, analysis of data, corrective and preventive action and management review.

All the crucial steps of the nano enabling process have been defined and the quality assurance requirements suitable for neFRP production were analysed through the review of the available standards for the production of composite and nano-enabled composite materials.

The main systematic actions of every nano-enabling manufacturing route are:

- **Resources** needed for company processes
- **Purchasing** of raw material
- **Production** of nano-enabled prepregs and composites
- **Measurement and improvement** of crucial steps
- **Health & Safety** issues during all the processes
- **Documentation** in order to ensure that quality objectives are met and traceability of the final product is possible.

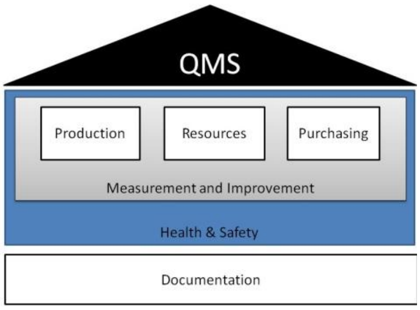


Figure 8. QMS Generic Graph illustrating the interactions between the main systematic actions

6.2 QA of manufacturing procedures

Two products have been identified for use in the nano-enabling of fibre reinforced polymers: nePrepreg and BuckyFilm. In order to achieve high quality products with the respective manufacturing procedures it is important that there are quality controls throughout the course of production processes for both materials and that after each processing stage the resulting products are checked before they proceed to the next one. Thus, for both processes, a total of four QA stages are implemented, from which the first two are common for the two processes.

A flowchart has been designed, illustrating the stages during which quality control of the products will be implemented and the quality parameters that have been identified for each of the production stages. The importance of the quality assurance of the different stages of the materials varies; QA of the final products is the most important and the importance lowers in the early stages of the procedure. The more important the quality assurance is, the more thorough the quality control of the product will be.

A plan to be implemented for the quality assurance of the manufacturing processes has been established for all of the QA stages. The QA plan will be implemented through the course of time and the techniques to be followed are classified in short-term (~ 1 year), mid-term (~ 3 years) or long-term (~ 5 years) planning.

7 Establishment of "Made to measure" process

The "Made to Measure" concept describes the steps to be followed in order to create something (material, process etc.) designed to fit a particular requirement/condition or solve a particular problem. "Made to Measure" materials provide multi-functionality tailored exactly to the customer's requirements. Under the framework of the NESTOR project the steps to be followed in order to tailor a composite material (FRP or nano-composite) to fit a specific requirement have been described.

In general, the "Made to Measure" process is based either on "trial and error" or on experience on material combinations that have been successfully used in the past. In the case of "trial and error", the experimental-based selection of a material for testing and prototyping may be time-consuming and expensive, given the amount of materials and respective combinations that will be investigated until the optimum scenario is selected, if feasible. However, the selection of a scenario based on previous experience may not always be realistic, since each customer has specific requirements that may have not been met before. Alternatively, there is the option of developing new materials to meet different requirements, but in this case the added cost and risk is very high.

Prior to the "Made to Measure" process, some preparatory steps need to be performed. Setting up a proper database of all the available materials and processes (resources), as well as the materials already produced by the company (heritage) is the most important step of the "Made to Measure" process, as it sets the foundation for a high quality analysis and selection. The following two types of databases are explained in the next paragraphs.

7.1 Steps towards "Made to Measure" materials

The strategy of the "Made to Measure" process is determined by the need to satisfy the requirement and involves three main phases and two CheckPoints (CKPT), as shown in Figure 9 below. The "Made to Measure" process is set with a goal to maximize the quality of the delivered product and minimize the cost and risk of the production process.

The overall process starts with the definition of the customer's requirements and finishes with the production and the provision of the product to the customer. Between these two, there are three phases: the Preliminary Phase, the Material Design & Development Phase and the Production Phase.

The exact steps towards "Made to Measure" materials are illustrated in Figure 9.

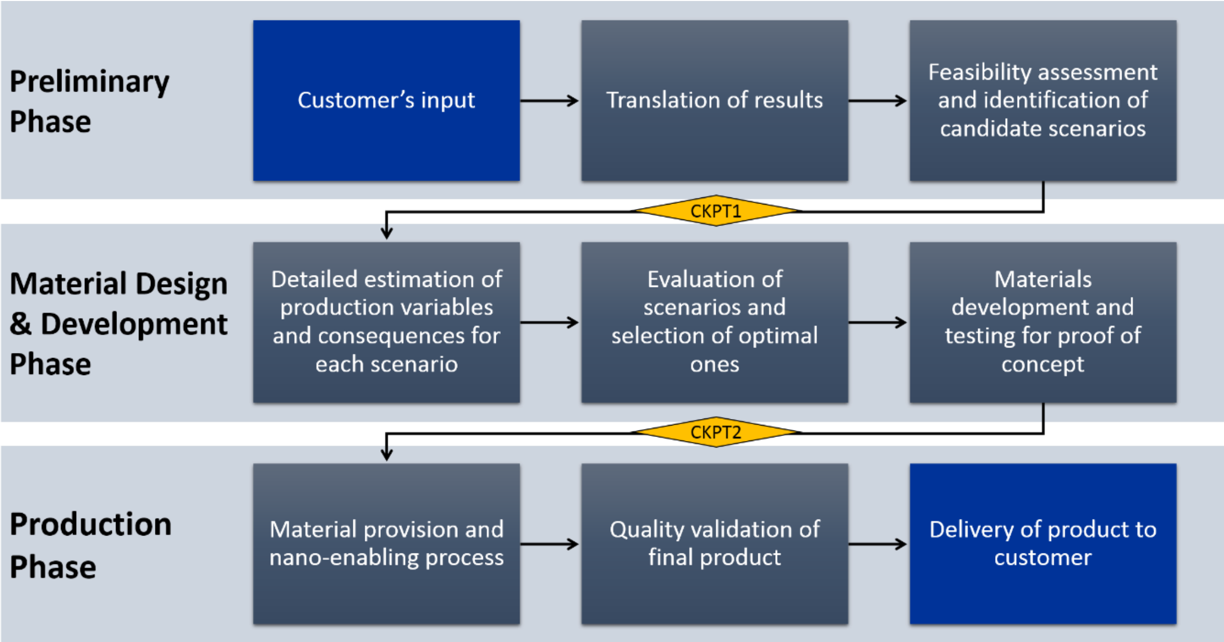


Figure 9. Illustration of end-to-end "Made to Measure" process. The process is structured in three phases: Preliminary, Material design and Production. Between each set of consecutive phases there is a CheckPoint where the company interacts with the customer and verifies the proceeding of the process.

During the **Preliminary phase**, the customer's requirements are registered, translated and analysed to a preliminary extent in order to assess the feasibility of the requested concept and roughly estimate directions towards viable production scenarios.

The end of the preliminary phase is established with **CKPT1**. During this CheckPoint, the company contacts the customer in order to verify, or not, the feasibility of the project and confirm that it will proceed to the Material Design phase. If the project is not considered feasible as is, the company may suggest alternative routes to achieve the desired results to the customer, until a solution is found.

After a solution has been found, the project proceeds to the **Material Design phase**. During this phase, all candidate scenarios that could yield the desired results are defined and investigated in detail with respect to the nano-enabling agent production variables and consequences (cost, time, risk etc.). Then, the candidate scenarios are evaluated, ranked and narrowed down to the optimal ones. Finally, an experiment is designed and executed to provide proof of concept for the selected scenarios.

When the company has concluded upon a preferable scenario, the company contacts again the customer during **CKPT2**. Then, the company informs the customer on the selected scenario, provides proof of concept and receives the approval of the customer to proceed to the Production phase. During this phase, the customer can interact with the company on the selection of the optimum scenario and rank with his personal criteria the evaluated scenarios. It is possible that a customer may pick a different scenario from the one suggested to him by the company if he decides to alter slightly his initial requirements in order to gain on something else. It must be noted that this CheckPoint is the final interaction point between the customer and the company before the delivery of the product, so all final decisions are made at that point.

Subsequently, the **Production phase** begins. During this phase the selected scenario proceeds to manufacturing and its quality is validated before it can be delivered.

8 Summary and Lessons Learned

NESTOR activity aimed to provide a strategic study for the use of neFRP materials in space industry. Towards this aim, different areas of industrial interest were enlightened to form a complete picture. Below are summarised some of the conclusions and achievements resulting from the NESTOR activity:

Selection of the most suitable nano-enabling techniques towards nano-enabled FRP products:

- After a detailed investigation of all the possible manufacturing routes and market potential was performed, the following nano-enabling techniques have been identified as the most suitable for the course of this project:
  - Integrating a *nano-doped resin film (FILM)* into the composite is a method that is expected to satisfy all space applications and industrialization criteria and is applicable both to Autoclave and RTM.
  - For Autoclave manufacturing, integrating nano-phase on the prepreg (nePrepreg) is also promising and can be achieved in three ways: by *powder scattering-coating*, by inserting nano-phase with the *lamination of nano-doped film* or by depositing a *nano-doped slurry*.
  - For RTM manufacturing, *inserting the nano phase into the matrix* or *veil technology* seems to satisfy the criteria set and can be considered good alternatives to *nano-doped resin film*.

Definition of Near Term Material Development Streams and initial material performance evaluation:

- The matching of nano-enabling processing technologies with Materials Development Streams resulted that for near-term (within NESTOR activity) autoclave composite production and ne-Prepreg (scattering) was the area of greater focus.
- Further to electrical performance, thermal and damping enhancement were selected as most promising material development streams (MDS).
  - 20 different formulations were tested for the aforementioned two new MDS.
- Materials treated with certain ceramic powders exhibited potential for improvement in thermal properties. Ceramic, carbon and hybrid concepts are selected as the most promising for further assessment in the future.
- Results for damping were inconclusive and a deeper investigation should be performed.

Quantification of expected impact

- A study was performed to investigate the possibility of utilizing nano-enabled composite materials for electronics enclosure. Results showed that an electronics housing made of a suitable neFRP can be comparable in terms of thermal performance and mass with an aluminum one.

Survey and Design of Quality Assurance system for nano-enabled FRP

- A quality management system was established for every aspect of the nano-enabling process and all relevant procedures, in accordance with the requirements of AS 9100, ISO, ECCS and other standards.
- A specific quality control plan for the company's manufacturing procedures has been identified and is scheduled to be implemented in the future.

"Made to measure" philosophy and new multifunctional solutions

- A 9-step procedure was established allowing the engineers to follow a stabilised procedure from the moment of the customer's input until the delivery of the product.