



AIRPOXY

Thermoform<u>a</u>ble, repa<u>i</u>rable and bondable sma<u>r</u>t e<u>poxy</u>- based composites for aero structures

Deliverable 1.1

Report on the preliminary specifications for demonstrators

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Glossary

Abbreviation / acronym	Description
AMGA	Horizon 2020 Annotated Model Grant Agreement
BVID	Barely visible impact damage
CA	Consortium Agreement
CFRP	Carbon Fiber Reinforced Polymer
DCM	Discontinuous Compression Moulding, process in which the material is hot formed / compression moulded in one or several discontinuous steps
DOA	Description of Action
EC	European Commission
Enduring prepreg	The term "enduring" prepreg means that the prepreg ply is partially or completely cured, so it does not need to be stored refrigerated. The 3R material is in this case proposed as a roll or sheets of (almost) cured individual plies. Thanks to the welding properties of the 3R materials, there is no time limitation between the manufacturing date of the prepreg and its processing date
F & DT	Fatigue and Damage Tolerance
FVC	Fiber Volume Content
GA	Grant Agreement
GFRP	Glass Fiber Reinforced Polymer
ILSS	Interlaminar Shear Strength
IM	Intermediate Modulus
IVW	Institut für Verbundwerkstoffe
LL	Limit Load
NDT	Non Destructive Testing
RF	Reserve Factor.
RTM	Resin Transfer Moulding
SQRTM	Same Qualified Resin Transfer Moulding
UL	Ultimate Load
UOI	University Of Ioannina
WP	Work Package





1. Executive Summary

This document compiles the specification required to define the two demonstrators that will be manufactured and tested during AIRPOXY Project, taking into account the needs to validate and demonstrate the thermoforming, bonding and repair technologies with 3R epoxy composites.

This means, on one hand, specifications regarding design. A basic design is developed generating some preliminary 3D models of all the sub-parts, taking into account the state of the art of the corresponding sub-elements design, possible geometrical constraints and interfaces. Preliminary thicknesses and lay-ups are also defined.

On the other hand, mechanical, physical, chemical and thermal properties are determined. This document just defines the requirements at sub-component level (geometrical stability and dimensional tolerances, maximum allowable defect based on structural requirements), whereas requirements at coupon level (i.e. required values for Tg, CAI, ILSS...) are defined in deliverable "D1.3 Report on preliminary analysis and definition of specifications for the raw materials".

Finally, the validation tests to check the repair and bonding technologies at sub-component level in WP5 will be defined too.





2. Introduction

This deliverable ("D 1.1. – Report on the preliminary specifications for demonstrators") is part of the specification of the AIRPOXY Project demonstrators and only specifications and requirements at sub-component level are provided (i.e. design, mechanical, physical, chemical and thermal properties and tests to be done in order to validate the bonding and repair technologies).

Two representative demonstrators are selected taking into account the needs to validate and demonstrate the thermoforming, bonding and repair technologies with 3R epoxy composites:

- a sub-component of a fan-cowl
- a sub-component of a leading edge

As far as the material specifications and the properties at coupon level are concerned, they are defined in deliverable "D 1.3 Report on preliminary analysis and definition of specifications for the raw materials". Besides, the master test plan is also included in this document. Regarding process requirements, deliverable "D 1.2 Report on the preliminary analysis and definition of process technologies requirements for manufacturing, bonding and repair" will be submitted.





3. Content

3.1. Sub-components of a Fan Cowl.

This demonstrator is part of a representative Fan Cowl door. Fan Cowl doors are one of most relevant parts of an engine nacelle. Typical configuration of major nacelle components is as per figure below:



Figure 1: Typical configuration of major Nacelle components.

Fan Cowl doors are composed of two curved CFRP stiffened panels hinged to pylon structure at their upper edge and attached together at lower edge line. The skin is reinforced by some longitudinal and transversal stiffeners (see Figure 2).







Figure 2: Typical assembly of the Fan Cowl door components (skin, stiffeners and hinges).

The demonstrator of AIRPOXY project will represent a section of the assembly of the three elements shown in Figure 2 (a skin, a transverse stiffener and a longitudinal stiffener) at the upper edge.

The main requirements to be taken into account would be:

- 1. The transverse stiffener must be used as support of the metallic hinges.
- 2. The whole structure must withstand the load introduction through the metallic hinge.
- 3. Geometric restrictions as there cannot be any interferences with other elements of the engine.
- 4. Weight must be optimized.
- 5. Design to cost.

Therefore, the preliminary design of the demonstrator will be as shown in Figure 3.









3.1.1. Sub-components definition.

Skin (s).

The skin will be a monolithic CFRP manufactured by IDEC in RTM (Resin Transfer Moulding) technology. Based on aeronautical requirements, it will be manufactured with an Intermediate Modulus (IM) dry 5HS fabric and a 58% of Fiber Volume Content (FVC).

The thickness of the skin normally is very close to 1 mm. However, the areas below the stiffeners are reinforced in order to avoid surface marks and allow a correct load distribution. These reinforced areas usually have a thickness between 2.5 and 4 mm.

In this case and in order to simplify the stiffeners geometry, the thickness of the skin will be constant and the panel will be flat. Panel size will be 500 x 460 mm, size enough to have a representative portion of each element. The preliminary thickness will be set to 2.95mm. For skin material reference, 0° direction is parallel to the circumferential direction of the Nacelle (see Figure 5). The final thickness and lay-up will we defined in WP5 based on a stress substantiation.



Figure 4: Demonstrator sub-element: skin.







Figure 5: Main dimensions of the skin.

Shape	L,s (mm)	W,s (mm)	Material	FVC (%)	t,s (mm)	s Lay-up
Flat	500	460	IM CF Fabric 3R Resin	58	2.95	(45/0/45/0/45/0)\$ (1)

⁽¹⁾ \$ means mid ply symmetric laminate (odd number of plies).

Table 3-1: Skin preliminary specifications.

Longitudinal Stiffener (Is).

The longitudinal stiffener will be a monolithic CFRP manufactured by ÉireComposites in Thermoforming. Based on aeronautical requirements, it will be manufactured with an Intermediate Modulus (IM) dry 5HS fabric and tape and a 58% of Fiber Volume Content (FVC).

The longitudinal stiffener will be designed as an omega shape in order to increase the torsional stiffness of the structure. A little pan-down will be done (section height variation) as this is something usual in this type of part. The head will be reinforced in order to carry the main loads (tension and compression loads), as it is in omega shape normal real parts in aircrafts. The thicknesses and lay-ups defined in Table 3-3 are preliminary and will be defined in WP5 based on a stress substantiation.



Figure 6: Demonstrator sub-element: longitudinal stiffener.





During WP2 trials it will have to be check if it is possible to achieve by the thermoforming process the pan-down and the different thicknesses. Based on those results, the longitudinal stiffener will be redesigned in WP5.

As shown in Figure 7, material reference 0° direction will be parallel to the circumferential direction of the nacelle.







Shape	L,ls	W,ls	w,ls-f	h1,ls	h2,ls	R,ls-h	R,ls-f	α
	(mm)	(mm)	(mm)	(mm)	(mm)	(mm)	(mm)	(°)
Flat omega	455	180	47	53	50	9	5	100

Table 3-2: Longitudinal stiffener	preliminary	dimensions.
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Material	FVC	t <i>,</i> ls-h	LS Head	t,ls-w & t,ls-f	ls Web & foot
	(%)	(mm)	Lay-up	(mm)	Lay-up
IM CF Tape IM CF Fabric 3R Resin	58	2.66	(45/0/45/0/ <u>0</u>) S ⁽¹⁾	2.15	(45/0/45/0)S ⁽¹⁾

⁽¹⁾ S means symmetric laminate (even number of plies).

Table 3-3: Longitudinal stiffener preliminary material, thicknesses and lay-ups.





Transverse Stiffener (ts).

The transversal stiffener will be a monolithic CFRP manufactured by IDEC in Resin Transfer Moulding (RTM) technology. Based on aeronautical requirements, an Intermediate Modulus (IM) dry 5HS fabric and tape and a 58% of Fiber Volume Content (FVC) will be used.

The transversal stiffener consists in a double T section beam that is installed in flight direction over the skin and longitudinal stiffeners with the scope of increase the stiffness in that direction. Therefore, the transversal stiffener of the demonstrator will be designed as crossing between an omega shape and a double-T shape profile. Besides and as explained before, it must be used as support of the metallic hinges (see Figure 2 for a typical hinge fitting design) that are fastened to the head and the foot of the omega profile. Therefore, a narrower cap must be designed in the upper flange at the edge close to the crossing area.



Figure 8: Demonstrator sub-element: transversal stiffener.

As shown in Figure 9, material 0° direction is defined as parallel to the circumferential direction of the Nacelle, and therefore, perpendicular to the beam axis.







Figure 9: Main dimensions of the transverse stiffener.

Shape	l,ts	w,ts-o	w,ts-dt	w <i>,</i> ts-o-f	w <i>,</i> ts-dt-h	h,ts-o	h,ts-dt	R,ts-o-h	R,ts-o-f	α
	(mm)	(mm)	(mm)	(mm)	(mm)	(mm)	(mm)	(mm)	(mm)	(°)
Flat Omega & Doble T	170	180	420	41	50	56	56	11	3	100

Table 3-4: Transversal stiffener	preliminary	/ dimensions.
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Material	FVC	t,ts-o-h	ts-o-h	t,ts-o-w & t,ts-o-f	ts-o-w & ts-o-f
	(%)	(mm)	Lay-up	(mm)	Lay-up
IM CF Tape IM CF Fabric 3R Resin	58	2.66	(45/0/45/0/ <u>0</u>)S ⁽¹⁾	2.15	(45/0/45/0)S ⁽¹⁾

⁽¹⁾ S means symmetric laminate (even number of plies).

 Table 3-5: Transversal stiffener omega preliminary material, thicknesses and lay-ups.

Material	FVC	t,ts-dt-h	ts-dt-h	t,ts-dt-w & t,ts-dt-f	ts-dt-w & ts-dt-f
	(%)	(mm)	Lay-up	(mm)	Lay-up
IM CF Tape IM CF Fabric 3R Resin	58	2.66	(45/0/45/0/ <u>0</u>) S ⁽¹⁾	2.15	(45/0/45/0)S ⁽¹⁾

⁽¹⁾ S means symmetric laminate (even number of plies).

Table 3-6: Transversal stiffener double-T preliminary material, thicknesses and lay-ups.





3.1.2. Bonding Of Elements.

The bonding of the three elements (skin, longitudinal stiffener and transverse stiffener) will be done by IDEC with a 3R film adhesive. The longitudinal stiffener flanges will be bonded to the skin while the transverse stiffener will be bonded to the longitudinal stiffener and also to the skin as shown in the figure below (Figure 10).



Figure 10: Position of the three elements.

The surfaces coloured in yellow in Figure 11 are the surfaces where the adhesive will be located.





The design of the different elements will consider the gap for the adhesive. This gap depends on the surface finish of the manufacturing tooling, so in this case, it will be set to ±0.1mm as all the elements will be manufactured in a close mould made of machined steel.

On the other hand, the adhesive film will have a thickness close to 0.3mm in order to assure the filling of the gap between the elements.

The bonding methodology and parameters will be defined in WP3.





3.1.3. Structural requirements definition.

Statistical basis to use in stress analysis.

Material strength data will be used in terms of B-basis values.

Material stiffness data (elastic moduli) will be used in terms of mean/averaged values. Exception is made only for composite materials in buckling analyses, where B-basis values shall be used instead.

Design Allowables derivation shall be made out of obtained test campaign data (see Material Requirements and Test Plan compiled in deliverable "D1.3 Preliminary analysis and definition of specification for raw materials").

In this stage of the material system development (TRL3) few coupons will be tested per property (compared to a Material Certification campaign). So, in case the dispersion of results obtained in the tests would be too high to consider B-basis values material strength data will be used in terms of mean/averaged values.

Damage Tolerance and detectability threshold.

For composite structure the following substantiation shall be provided by analysis, supported by test, for the different damage types according to certification principles.

The residual strength of the structure shall be adequate to carry ultimate load during the whole Design Life Goal with BVID damages present in the structure. Design allowable values shall consider a barely visible impact damage (BVID) where dent depth shall be at least 0.3 mm after relaxation for an impact energy not higher than 35 Joules (energy cut-off).

Expected standard classification of defect severity during production in CFRP laminates will correspond to a maximum indication area during Non-Destructive Testing (NDT). Minimum BVID area to be considered in the definition of damage tolerance allowable values will not be lower than 500 mm².

Environmental conditions.

Certification regulations require the composite structures to be substantiated for the most adverse environmental conditions (including the extremes of conditions) encountered within the airplane operating envelope. Experience has shown that strength properties of carbon fiber materials (especially those resin dominated) are worst degraded in an ambient with moisture and elevated temperature.

The design service temperature for Fan Cowl structural justification is 120°C. An environmental scenario of material moisture saturated at 70°C/85%RH until equilibrium and working at 120°C is then assumed (H/W4).





Static strength (and residual strength) requirements.

The applicable requirement for this structure, as an assembly of composite elements in a Fan Cowl, would be to demonstrate that the structure withstands:

– Ultimate Loads (UL) without failure.

In this kind of parts, composite residual strength analysis shall be performed considering BVID damages at UL level.

Fatigue and Damage Tolerance requirements.

Structural design life under normal operational conditions shall be of 75000 flights cycles (Design Service Goal, DSG) with an average flight time being 120 minutes with an initial inspection (threshold) at 15000 flight cycles and a repetitive inspection interval of not less than 7500 flight cycles.

For fatigue analysis the requirements are no crack initiation during the full aircraft Design Service Goal (DSG=75000FC) and demonstrating the threshold objective.

For composite parts, the analysis could be supported by existing specific tests on similar structural design concepts. Including artificial defects and damages in detail/subcomponent fatigue tests will substantiate it. The initiation nongrowth concept on composite parts will be guaranteed by limiting an adequate maximum allowable static strain, based on the experience of aircraft manufacturers. Including artificial defects and damages in detail/subcomponent fatigue tests will substantiate it. However, at this stage of TRL no Fatigue and Damage Tolerance test of the demonstrator will be done, as it is far away from the objectives of the project: to validate and demonstrate the thermoforming, bonding and repair technologies with 3R epoxy composites.

For Damage Tolerance the residual strength is adequate to carry ultimate load during the whole Design Life Goal with this kind of damages present in the structure:

- Barely Visible Impact Damages (BVID) for energy not higher than 35J
- Allowable manufacturing defects
- Allowable in-service damages





Load definition.

Out of the different tests results from test campaign, a preliminary design allowables derivation would be done to proceed with substantiation, then covering first step along the path for certification. Therefore, the demonstrator elements will be sized based on a Static and Fatigue and Damage Tolerance (F&DT) preliminary assessment.

Static loading.

For static loading, most critical case is the one coming from bending/shear of the transversal section of the hinge fitting (see Figure 12).



Figure 12: Hinge fitting loading.

Critical Flight Load cases (Limit and Ultimate conditions) to be considered are:

Flight Loading

- Maximum in-plane Limit Load:
 - Fyz = 22000 N (Angle α = -5º)

- Maximum in-plane Ultimate Load:

Fyz = 31000 N (Angle α = -5°)

Summarizing,

	Flight Loading		
Condition	LL	UL	
Type of Load	Max in-plane	Max in-plane	
Load F (N)	22000	31000	
Angle α (°)	-5	-5	

Table 3-7: Hinge Fitting critical loading.





Fatigue loading.

At this stage of TRL no Fatigue and Damage Tolerance test of the demonstrator will be done, as it is far away from the objectives of the project: to validate and demonstrate the thermoforming, bonding and repair technologies with 3R epoxy composites.

3.1.4. Quality and Dimensional requirements.

Dimensional requirements

There is a shape/flatness requirement of \cap = ± 0.2 mm at the external surfaces (upper and lower flanges or cappings). This tolerance is related to the process technology (close mould made of steel).





Figure 13: Longitudinal Stiffener: surfaces with shape/flatness requirement.



Figure 14: Transverse Stiffener: surfaces with shape/flatness requirement.

On the other hand, there is a trimming boundary line requirement of ± 0.5 mm.



Figure 15: Trimming boundary line for longitudinal and transverse stiffeners.

Thickness tolerances are also related to the process technology. In this case \pm 0.3 mm value is defined.





Maximum allowable defect (porosity, delamination and wrinkles).

The quality of the sub-elements will be assessed through NDT (A-Scan, Pulso – eco method).

Defects acceptance criteria is based on the structure category. The longitudinal stiffener category is higher than the category of the transverse stiffener. However, the transverse stiffener at the crossing with the longitudinal stiffener is assumed to have the same category as the longitudinal stiffener.

Therefore, for defects in solid laminates except for the ones located at edges or radius:

	Longitudinal Stiffener & Transverse Stiffener At crossing	Transverse Stiffener	
Maximum area (mm2)	250	500	
Maximum length (L) (mm)	27	39	••••••

Table 3-8: Defects acceptance criteria for solid laminates except for the ones located at edges or radius.

For defects in solid laminates located at edges:

	Longitudinal Stiffener & Transverse Stiffener At crossing	Transverse Stiffener	L
Maximum area (mm2)	220	500	P
Maximum length (L) (mm)	22	30	
Maximum depth (P) (mm)	22	30	•••••••

Table 3-9: Defects acceptance criteria for solid laminates located at edges.

For defects in solid laminates located at radii:

	Longitudinal Stiffener & Transverse Stiffener At crossing	Transverse Stiffener	P
Maximum area (mm2)	200	500	
Maximum length (L) (mm)	30	50	
Maximum depth (P) (mm)	24	41	

 Table 3-10: Defects acceptance criteria for solid laminates located at radius.





Porosities with an attenuation lower than 18dB are not considered as defects.

As far as the wrinkles concerns, wrinkles up to 0.25mm and length up to 25mm in a maximum area of 30cmx30cm will be accepted without any correction.

3.1.5. Sub-component test.

Bonding validation.

One Fan-Cowl demonstrator will be produced for bonding validation in WP5. Lap Strap tests will be performed on some coupons extracted from the demonstrator glued areas (see location of coupons in Figure 16).



Figure 16: Extraction of coupons for bonding validation.

Lap Strap is not a standard test as it is not a method for deriving data. A typical lap shear test would be more appropriate, but the required coupon geometry cannot be obtained from the demonstrator geometry. As the same coupons are made from flat panels and tested in WP4, those values could be compared with the ones obtained from the demonstrator in order to validate the bonding technology.

Besides, a full scale C-Scan of the demonstrator at the final stage of the project could be done (subcontracted by UOI).





Repair validation.

To validate the repair technology in WP5, one transverse stiffener will be manufactured, afterwards damaged (delaminated) and finally repaired.

In order to create a delamination, an unfolding of one of the radii of the transverse beam at the crossing will be done as shown in the following figure:



Figure 17: Flexural test to create a delamination at one fillet radius.

Once the delamination is repaired and checked by Ultrasonic inspection (A-Scan, Pulso – eco method), the same flexural test will be done to confirm an equivalent behaviour to the undamaged one.



3.2. Sub-components of a Leading Edge.

3.2.1. Sub-components definition.

SONACA's demonstrator is a wing leading edge representative of commercial airplane parts. The demonstrator is a shorter (with a maximum span-wise dimension around 2.2m) and simplified version, composed of three elements: one skin and two webs.



Figure 18: Leading-edge.

- The leading-edge skin is a monolithic composite part that will be manufactured in SQRTM (Same Qualified Resin Transfer Molding). Its shape is globally conical with very little double curvature.
- The two webs are monolithic CFRP fabric parts.



Figure 19: Demonstrator of the Leading-edge.





Leading-edge SKIN.

Baseline lay-up.

The base-line lay-up of the leading-edge skin will be made of the following layers (see Figure 20):

- **External metallic facing**. This 0.4mm metallic sheet is co-bonded with the rest of the lay-up during the SQRTM process and is here as a protection against erosion.
- **Hybrid lay-up.** This hybrid lay-up is composed of structural CFRP Uni-directional plies, dielectric GFRP fabric plies and an embedded electrical resistance for the de-icing.
- Internal surface 3R adhesive film. The 3R adhesive ply is co-cured with the rest of the lay-up during the SQRTM process.
- Internal metallic facing. This 0.4mm metallic sheet is identical to the external one, with openings where the webs are bonded.



Figure 20: Details on the baseline lay-up of the skin.





"Specific case" lay-up.

In some specific cases, the lay-up may vary, with the removing of the metallic facings and the addition of an expanded metal foil on the external surface. This secondary lay-up will be made of the following layers:

- **Erosion coating:** Instead of the metallic facing, a specific coating may play the role of the erosion protection. Its thickness would be around 0.2 mm. This coating would be a dielectric.
- Expanded metal foil: An expanded metal foil is then embedded at the surface of the dielectric plies.
- **Hybrid lay-up.** This hybrid lay-up is composed of structural CFRP Uni-directional plies, dielectric GFRP fabric plies and an embedded electrical resistance for the de-icing.
- Internal surface 3R adhesive film. The 3R adhesive ply is co-cured with the rest of the lay-up during the SQRTM process.



Figure 21: Details on the "specific case" lay-up of the skin.





Leading-edge WEBS.

The two webs are made of CFRP + GFRP fabric with 3R resin. Each web is made of two half parts that will be thermoformed independently. The two webs (and therefore the four half webs) are different from each other due to the conical shape of the leading-edge skin.

Each half-web shall be made of 4 carbon fabric plies: [0°; 45°; 90°;-45°] the -45° ply being the external one.

The approximate thickness of each half web will be around 1.25 mm.

The space between the two half-webs and the skin shall be filled by noodle filler.



Figure 22: Definition of the webs.

Due to the double curvature of the demonstrator, the web thermoforming can show problems that would have to be analyzed in WP2.

The assembly process between both halves of the web must be also analyzed.





3.2.1. Bonding Of Elements.

After the thermoforming of the four half-webs, they shall be assembled with the noodle fillers in order to form complete webs. Following this, the two webs will be welded with the skin as part of the final process.



Figure 23: Induction welding area of the webs to the skin. Leading edge demonstrator.

The webs are manufactured with 3R material and the skin is manufactured with a 3R adhesive layer on its internal face. The objective is to weld them by providing enough heat and pressure so that the 3R material of each substrate can fuse and form covalent bonds. The development of the welding will be carried by IVW in WP3 and transferred to EIRE in WP5 for the assembly of the webs and the final assembly of the webs and the skin.





3.2.2. Quality and Dimensional requirements.

Quality requirements.

After manufacturing (SQRTM for the skin and Thermoforming for the webs), the quality of the sub-elements will be assessed through NDT and thickness measurements:

- NDT. The parts will be inspected in Ultra-sound Pulse-Echo. The maximum acceptance limit for the porosities will be 9 dB of attenuation for the webs and 12 dB for the skin.
- Thickness. The requirement for thickness is -8% / +10%.

A control process panel will be injected with the skin in order to obtain ILSS control process specimens to assess the quality of the curing/injection.

Dimensional requirements.

The dimensional requirements are:

- +/- 0.8mm on the overall external surface of the skin.
- +/- 0.3mm on the overall external volume of the webs.

The skin and the webs shall be controlled BEFORE & AFTER their welding together.





3.2.1. Sub-component test.

Bonding validation.

Two demonstrators will be manufactured for bonding validation.

The first demonstrator will be impacted at four locations (two per web) along the bondline between the skin and the webs to assess the delaminations. The impacts (normal to the surface) will be performed on a tool already available at SONACA. Results will be compared to already available reference values.



Figure 24: Impacts on leading edge #1.

Pull out specimens will be extracted from the second demonstrator to assess the final bonding performance. Test results will be compared to reference values and to the flat panels pull-out specimens of WP3.2.3.



Figure 25: Pull-out tests on leading edge #2.

Repair validation.

In order to validate the repair procedure, the impacted Leading edge #1 will be sent back to EIRE for repair. Pull out specimens will be extracted from the repaired web/skin bondline and tested in order to be compared with pull-out tests from leading edge #2.





4. Conclusion.

This document is a preliminary specification of the two demonstrators that will be manufactured and tested during AIRPOXY Project, taking into account the needs to validate and demonstrate the thermoforming, bonding and repair technologies with 3R epoxy composites.

Specifications regarding design and mechanical, physical, chemical and thermal properties at sub-component level are defined as well as the validation tests to be done at sub-component level to check the repair and bonding technologies.

For both the Fan-Cowl and the Leading-Edge demonstrators, the final thicknesses and lay-ups of all the elements will be defined in WP5 based on basic stress substantiation.

Besides, during WP2 trials, it will have to be checked if it is possible to achieve with the thermoforming process and the material system selected (3R Resin + FC fabric and tape) the shape and the thicknesses suggested for both demonstrators. As a result, at the end of the project, a final report collecting the final specifications will be submitted (deliverable "D1.4 – Report on the final specifications for demonstrators, process technologies and raw materials").