Multifunctional structures with quasi-solid-state Li-ion battery cells and sensors for the next generation climate neutral aircraft

Horizon Europe | HORIZON-CL5-2021-D5-01-05

Greenhouse gas aviation emissions reduction technologies towards climate neutrality by 2050

D4.1 Smart cell/Structural manufacturing process report

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PROJECT ABSTRACT

MATISSE responds to the fourth bullet of the HORIZON-CL5-2021-D5-01-05 topic “expected outcome”, delivering improved aircraft technologies in the area of multifunctional structures capable of storing electrical energy for hybrid electric aircraft applications. This consists in integrating Li-ion cells into aeronautical composite structures, sharing the load-bearing function with the structure and achieving an aircraft structural element capable of functioning as a battery module.

To do so, MATISSE will:

- advance Li-ion battery cell technology, in a non-conventional formulation suitable for bearing structural loads: NMC811 (cathode), Si/C (anode) and bicontinuous polymer-ionic quasi-solid-state electrolyte (BCE), i.e. NMC811|BCE|Si/C, achieving 170-270 Wh/kg at cell level;
- enable the functional integration of Li-ion cells into solid laminate and sandwich composite structures;
- make the structural battery smart, by equipping it with on-cell and in-structure sensors, connected to a chip-based CMU (Cell Monitoring Unit) and PLC (Power Line Communication).

MATISSE delivers a multifunctional structure demonstrator capable of power delivery, power management and safety monitoring. This consists of a full-scale wing tip (1.42 m × 0.69 m) for use in place of the current wingtip assembly installed on Pipistrel Velis Electro, embedding a module of 40 battery cells at 72 VDC. This will undergo a comprehensive testing and characterisation campaign, qualifying the technology at TRL 4 at the end of the project (2025). MATISSE will also encompass aspects related to flight certification, life-cycle sustainability and virtual scale-up, paving the way towards the application of structural batteries as an improved performance key enabling technology for next generation commuter and regional hybrid electric aircraft applications.

The strong and complementary consortium of 8 partners from 5 different European countries and one associated partner country representing industrial companies, SMEs and RTOs is coordinated by AIT Austrian Institute of Technology. MATISSE is scheduled to run from September 1st 2022 to August 31st 2025, for a total duration of 36 months and has received funding from the European Union’s Horizon Europe research and innovation programme under Grant Agreement no. 101056674. A full list of partners and funding can be found at: https://cordis.europa.eu/project/id/101056674.

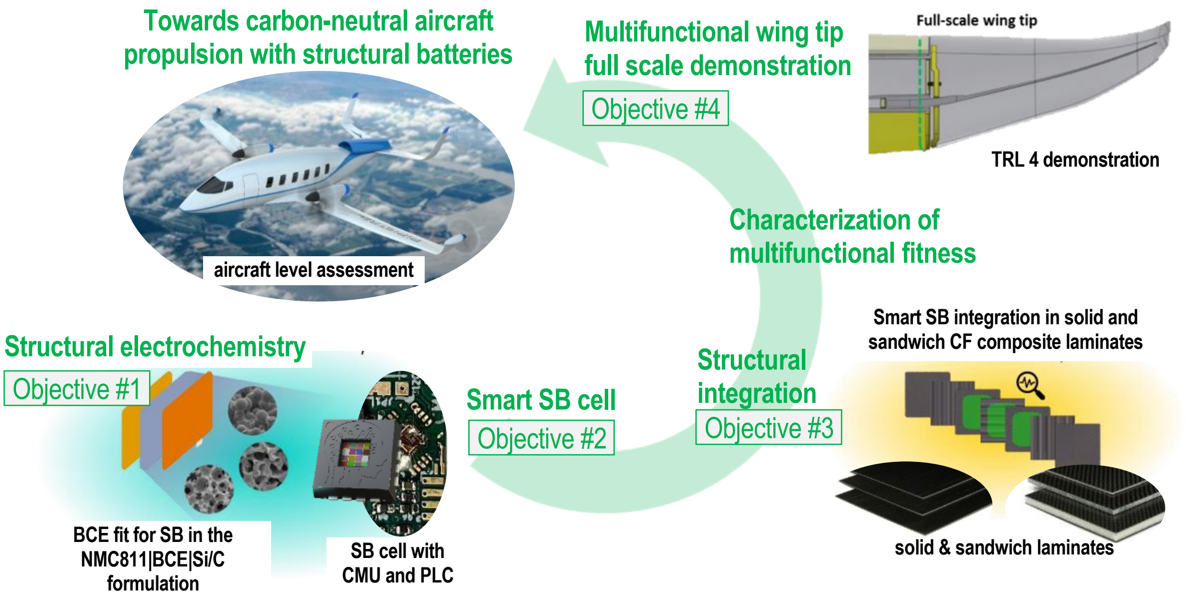

Figure 1: MATISSE concept overview (graphical abstract)

EXECUTIVE SUMMARY

This report provides the details about the choice of the composite material which will be considered in the MATISSE project to (a) preserve the electrical performances of the battery cells developed by AIT, (b) enable an easy introduction of embedded SHM controls within the battery cells and within the composite material, and (c) ensure the manufacturing constraints imposed by Pipistrel (out-of-autoclave process) in order to obtain a technically and economically solution to produce the final demonstrator of the project.

At the beginning of the project MATISSE, it was demonstrated that the material considered in SOLIFLY project (and initially planned to be also used in MATISSE), despite many advantages, is not suitable considering an out-of-autoclave process. Therefore, it was decided to change the initially planned material to another one. A literature survey was performed, mainly by ONERA and Pipistrel, to determine potential alternative candidates. Moreover, technical meetings with experts from *Hexcel* and *Composite Distribution*, major composite material providers, to obtain additional information about the current composite material market and to be sure that the chosen solution could be applied in aeronautical industries in few years. Five carbon/epoxy composite materials have been studied in detail, and finally the IM2C/M79 composite material made of unidirectional fibres with an intermediate weight areal has been selected. Furthermore, the characterization test campaign was adapted in order to perform tests mandatory to prepare the final test on the MATISSE demonstrator.

1. INTRODUCTION

The choice of the composite material in MATISSE, to manufacture the demonstrator at the end of project, is a key aspect that has been addressed since the beginning of the project. Indeed, there are several constraints to consider in this choice in order to obtain: (i) an interesting trade-off between the mechanical performances and the associated cost of the material, (ii) a limited number of characterization tests to save time and money in the project, (iii) to preserve the electrical performances of the battery cells by considering a low temperature curing cycle, (iv) to obtain a good adhesion between the composite material and the battery cells for limiting the decrease of the residual properties and finally (v) to obtain a good isolation from an electrical point of view between the battery cells and the surrounding composite material to exploit the full potential of the cells. This report presents all these considerations in order to choose the best material to produce effective composite parts with embedded battery cells.

2. CHOICE OF THE COMPOSITE MATERIAL

2.1. AS4/8552 COMPOSITE MATERIAL (SOLIFLY PROJECT)

Initially, it has been chosen to consider in MATISSE the same carbon/epoxy material than was used in SOLIFLY project, which is the AS4/8552 unidirectional plies with an intermediate weight areal. Many publications in the scientific literature consider this composite material [1–5], and all the basic material properties (elastic properties, strengths) are available. Moreover, other properties can be found in the literature, such as the strength of open-hole plates subjected to tension and compression [1], or impact at different low speed/low energy levels and compression after impact [5], as illustrated respectively in Figure 2 and Figure 3.

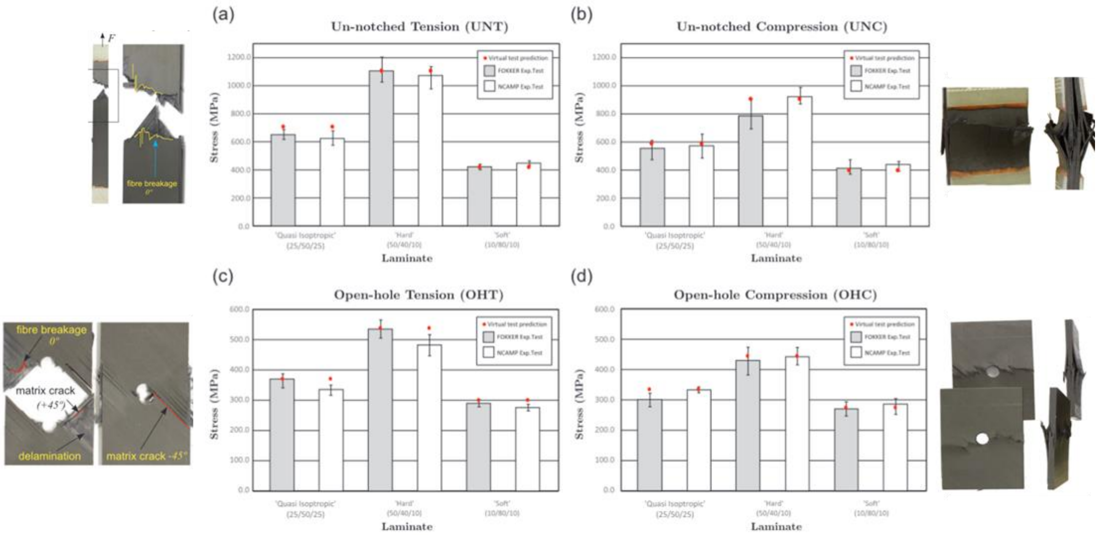

Figure 2: Test results on open-hole plates manufactured in AS4/8552 composite material [1]

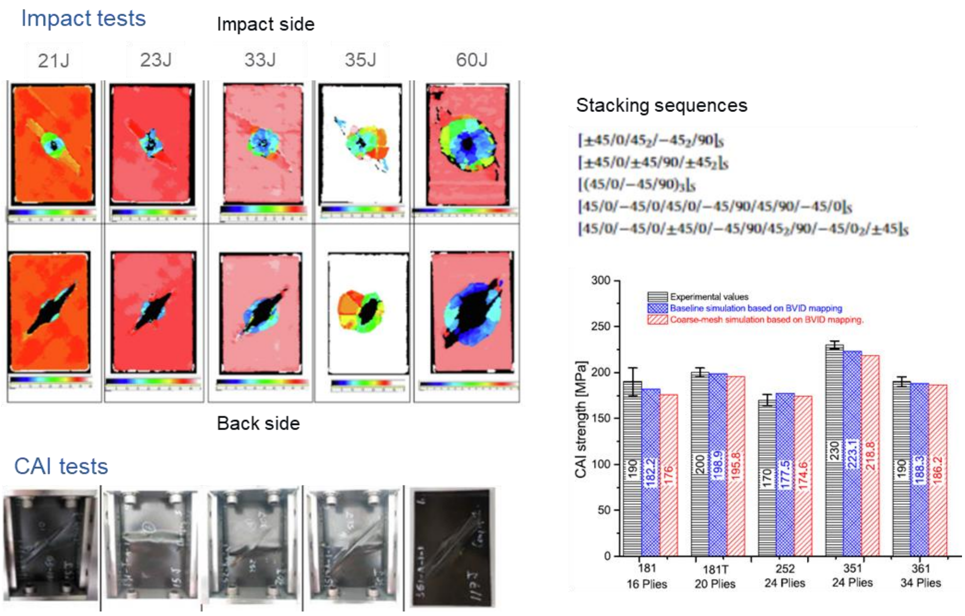

Figure 3: Impact tests and compression after impact tests on plates manufactured in AS4/8552 composite material [5]

This material, widely used in aeronautics, presents high mechanical properties. The mechanical properties usually used in advanced damage models, such as that developed at ONERA, are provided in Figure 4.

Property	Test Standard	Specimen number: 6 Mean (CV %)
Ply elastic properties		
E_{1t}(GPa)	ASTM D3039M [30]	137.1 (1.4)
E_{1c}(GPa)	ASTM D3410M [31]	114.3 (0.9)
E_{2t}(GPa)	ASTM D3039M [30]	8.8 (0.3)
E_{2c}(GPa)	ASTM D3410M [31]	10.1 (0.8)
$G_{12} = G_{13}$(GPa)	ASTM D3518M [32]	4.9 (0.8)
$\nu_{12} = \nu_{13}$	ASTM D3039M [30]	0.314
ν_{23}		0.487
Ply strengths properties		
X^T(MPa)	ASTM D3039M [30]	2106.4 (8.2)
X^C(MPa)	ASTM D3410M [31]	1675.9 (5.2)
Y^T(MPa)	ASTM D3039M [30]	74.2 (6.3)
Y^C(MPa)	ASTM D3410M [31]	322.0 (1.7)
S^L(MPa)	ASTM D2344M [34]	110.4 (1.3)
Thermal expansion coefficient		
α_1(°C−1)		$0.21e^{-06}$
α_2(°C−1)		$3.30e^{-05}$
Ply fracture energies		
$\mathscr{G}_{1+}$(kJ/m^2)	Pinho et al. [39]	125.0
$\mathscr{G}_{1-}$(kJ/m^2)	Catalanotti et al. [41]	61.0
$\mathscr{G}_{2+}$(kJ/m^2)	ASTMD5528 [42]	0.30
$\mathscr{G}_6$(kJ/m^2)	ASTMD7905 [43]	0.87
Interface properties		
τ_3^0(MPa)	ASTM D3039M [30]	74.2
τ_{sh}(MPa)	ASTM D2344M [34]	110.4
$G_{Ic,\theta=0°}$(kJ/m^2)	ASTMD5528 [42]	0.30 ± 0.01
$G_{IIc,\theta=0°}$(kJ/m^2)	ASTMD7905 [43]	0.87 ± 0.06
η_{bk}		1.45

Figure 4: AS4/8552 material properties found in the literature [1]

Moreover, its manufacturing process is quite flexible [6] and can be widely modified without degrading the mechanical properties. Indeed, in the SOLIFLY project, the cure cycle (considering the autoclave process) has been adapted by ONERA in order to preserve the electrical functionality of the embedded cells. The maximal cure temperature has been decreased from 180°C to 130°C, but this modification has induced a large increase of the duration of the cure cycle (from 2 hours to 18 hours), decreasing its potential interest for industrial partners. The pressure has been decreased from 7bar to 2.5bar to avoid leakage of the ionic liquid introduced in the electrolyte of the battery cells. In SOLIFLY, this manufacturing process has been successfully validated by producing laminated coupons and also a stiffened panel with several functional battery cells, as illustrated in Figure 5.

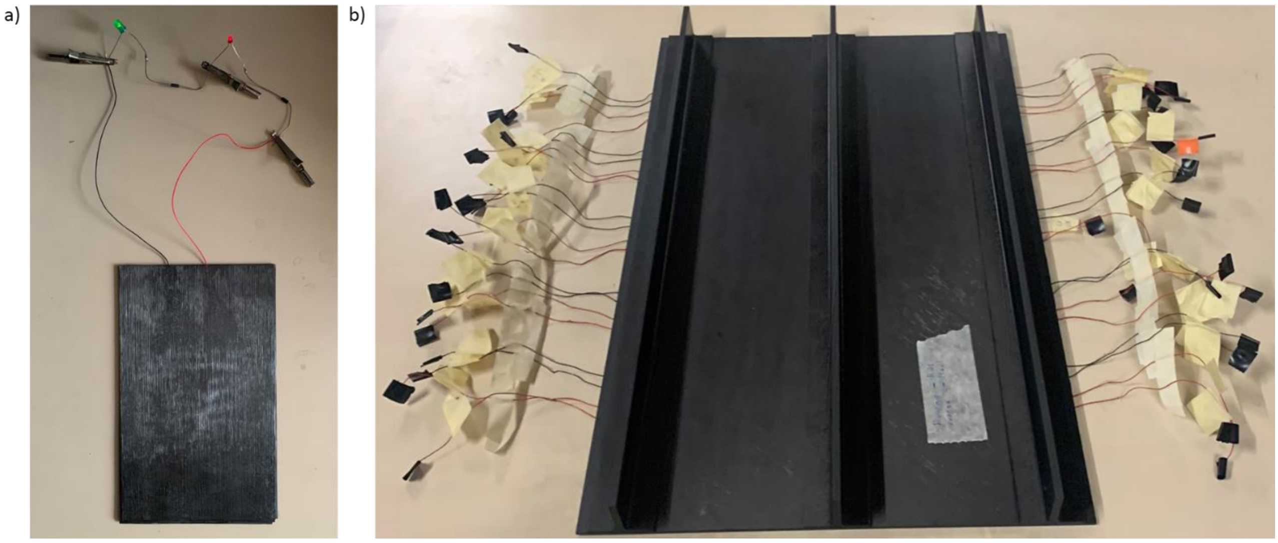

Figure 5: Functional cross-ply laminate with two battery cells and functional quasi-isotropic laminated stiffened panel with 20 battery cells produced in SOLIFLY project.

However, the use of standardized manufacturing procedures is one of the MATISSE project pre-requisites considering out-of-autoclave processes without applying any pressure to the composite material to limit the production cost. Therefore, as suggested by Pipistrel, an AS4/8552 quasi-isotropic composite plate has been manufactured without any pressure (except that applied by the vacuum bag) at ONERA. Figure 6 presents a micrograph of a polished free edge, where many voids are clearly observed. Considering a binarization method [7] of this micrograph, it is possible to determine the surface void content which is estimated around 22%, what is far higher that the threshold accepted in aeronautics (evolving usually between 0.5% or 3%).

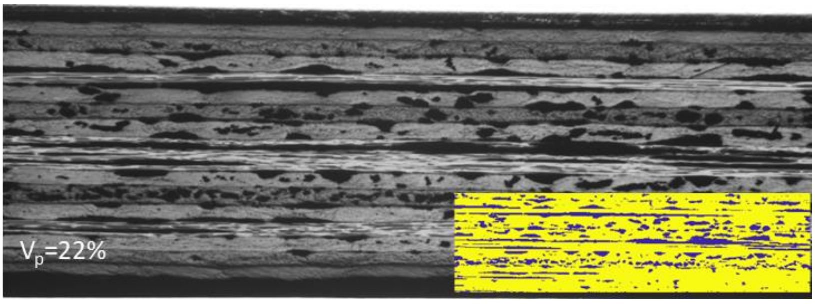

Figure 6: Analysis of one free polished edge of an AS4/8552 composite plate manufactured without any additional pressure

Therefore, considering such a manufacturing constraint (out-of-autoclave process to limit the production cost), it has been decided to change the composite material studied in MATISSE project. Pipistrel and ONERA have thus performed a literature survey about other potential candidates available in the market.

2.2. FIRST ALTERNATIVE CANDIDATE: IM7/M20 MATERIAL

Firstly, Pipistrel has identified the carbon/epoxy IM7/M20 material as a potential candidate, produced by Hexcel, because no additional pressure is needed (only the vacuum bag pressure) and the maximal cure temperature is fixed at 130°C. A literature survey has been performed in order to evaluate the potential of such a material. The mechanical properties have been found on the Hexcel website and are reported in Figure 7. The mechanical properties are similar than those of AS4/8552 composite material for tensile loadings, but significantly lower for compression loadings.

Test	Units	M20/40%/G904 (1)	M20/34%/134/IM7 (12K) (2)
Glass Transition Temp. (extrapolated onset E') - Dry	Method	EN 6032	
	°C	155 (1)	
	(°F)	(311 (1))	
Tensile Strength - warp RT test	Method	SACMA 4R-94	EN 2561B
	MPa	877	2790
	(ksi)	(127)	(405)
Tensile Modulus – warp RT test	GPa	65	175
	(ksi)	(9)	(25)
Tensile Strength – weft RT test	Method	SACMA 4R-94 869	-
	MPa	869	-
	(ksi)	(126)	-
Tensile Modulus - weft RT test	GPa	65	-
	(msi)	(9)	-
Compression Strength - weft RT test	Method	SACMA 1R-94	-
	MPa	840	-
	(ksi)	(122)	-
ILSS – warp RT test	Method	SACMA 8R-94	EN 2563
	MPa	78	110
	(ksi)	(11)	(16)
ILSS – warp 80°C (175°F) test	Method	SACMA 8R-94	-
	MPa	60	-
	(ksi)	(9)	-
In-plane Shear Strength RT test	Method	SACMA 7R-94	EN 6031
	MPa	110	120
	(ksi)	(16)	(17)
In-plane Shear Strength 120°C (250°F) test	Method	SACMA 7R-94	-
	MPa	80	-
	(ksi)	(11)	-

Figure 7: Mechanical properties of the IM7/M20 composite material

Nevertheless, very few data are available in the literature. Indeed, this material is produced only in Germany by Hexcel and has been mainly designed for composite repairing [8–10], as illustrated in Figure 8. Indeed, the flexible manufacturing process associated to this material is very relevant for composite repairing. The obtained residual strength is also very promising because this material presents a good adhesion with other composite materials or adhesives.

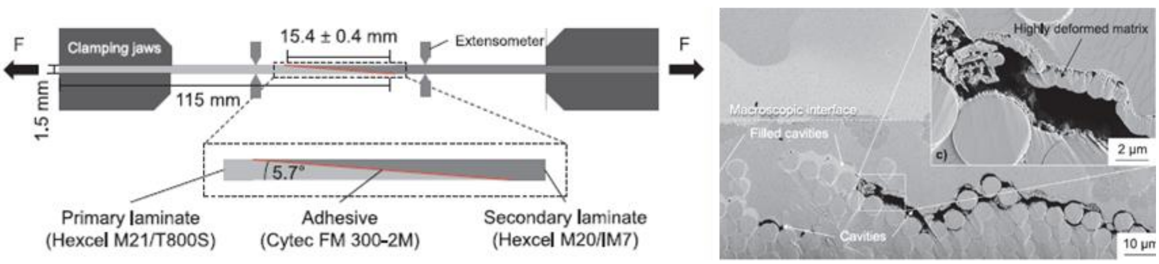

Figure 8: Composite repairing with IM7/M20 material which presents a flexible manufacturing process [9]

Technical meetings have been organised by ONERA with one composite material expert in *Hexcel* (M. Bonnafoux) who confirmed that the IM7/M20 composite material is produced only for repairing issues and only low material quantities are available. Therefore, it is not relevant to manufacture a large composite structure, as the winglet planned in the MATISSE project, with this composite material.

2.3. SECOND ALTERNATIVE CANDIDATE: IM7/M56 MATERIAL

Then, Hexcel has proposed to consider the IM7/M56 carbon/epoxy material, which is the composite material really designed by Hexcel for aeronautical industries using an out-of-autoclave process.

ONERA has thus performed a literature survey, and many publications dealing with this composite material have been found [11–16]. The void content has been considered using CT-scan [11] to determine precisely their size and their distribution and compared with those obtained on similar composite materials manufactured with an autoclave process, as reported in Figure 10. The quality of the produced coupons clearly fulfils the aeronautical standards.

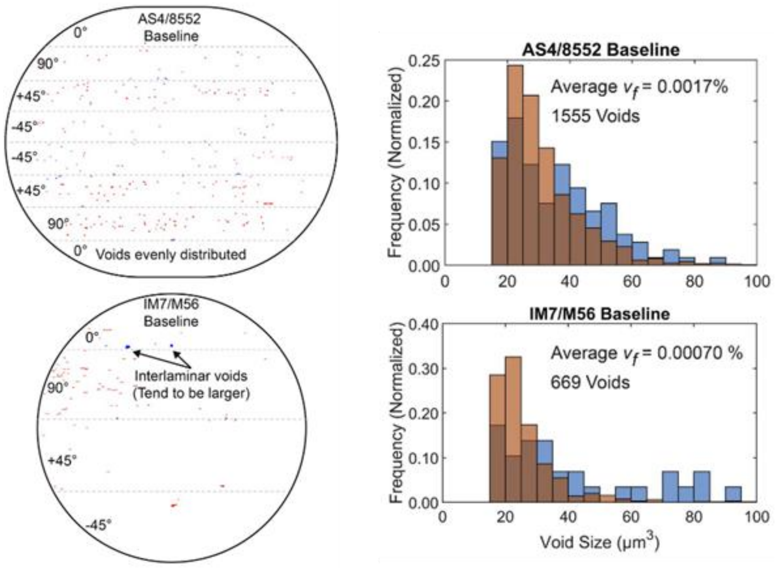

Figure 9: Study on initial voids in IM7/M56 material [11]

Moreover, large composite parts have been manufactured with this material and have already been presented at JEC Paris few years ago, demonstrating the feasibility to produce large parts with IM7/M56 material. Another interesting point concerns the possibility of recycling associated with matrix M56 as reported in [12,13]. The mechanical properties are rather similar to those of the AS4/8552 composite material which is manufactured with an autoclave process and has been considered in the SOLIFLY project, as reported in Figure 10. The mechanical properties are very high and fulfils the aeronautical recommendations.

Moreover, the influence of a change in the manufacturing process has been studied on the mechanical properties, which remains low by changing the maximal curing temperature (decreasing the temperature) as reported in [15]. The recommended curing cycle considers a maximal cure temperature fixed at 180°C. As in SOLIFLY project, with AS4/8552 composite material, the cure temperature could be decreased to 130°C to preserve the electrical performances of the battery cells, without decreasing the mechanical properties. Nevertheless, the decrease of the cure temperature will increase the duration of the cure cycle and this point could be an issue in order to transfer the developed methodology to aeronautical industries. This last point pushed Pipistrel and ONERA to consider another material.

Moreover, the cost of the IM7/56 material is quite expensive and does not fit with the recommendations to produce competitive composite parts.

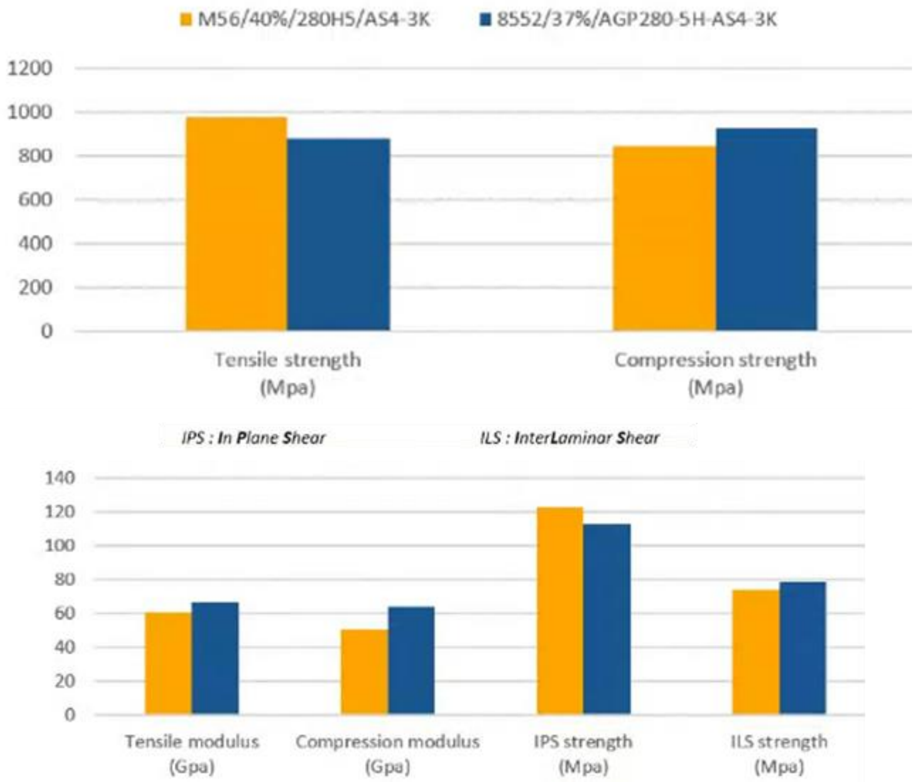

Figure 10: Comparison of the mechanical properties of IM7/M56 (out-of-autoclave) and AS4/8552 (autoclave) materials

2.4. THIRTH ALTERNATIVE CANDIDATE: TR50/M90 MATERIAL

The *Composite Distribution* company, which is the official French seller of Hexcel's products in France, also advised a new carbon/epoxy composite material, named TR50/M90. Indeed, Onera has organised a visio-conference to exchange on this composite material with J. Renard, a material expert in *Composite distribution*. This material does not need additional pressure, the maximal curing temperature is 110°C, the mechanical properties are moderate, as illustrated in Figure 11, but remain suitable for the manufacturing of the demonstrator in the MATISSE project. Moreover, Hexcel has developed a composite material for aeronautical industries, meaning that the quality of the material is guarantee for the next few years.

Test	Property	Units	Test Method	Test Direction	Conditioning	Test Temp. (°C)	Typical Performance
Tensile	Strength	MPa	EN 2561 Type A	0°	Dry	23	2400
Tensile	Modulus	GPa	EN 2561 Type A	0°	Dry	23	140
Compression	Strength	MPa	EN 2850 type B1	0°	Dry	23	1300
Compression	Modulus	GPa	EN 2850 type B2	0°	Dry	23	115
Interlaminar Shear	Strength	MPa	EN 2563	0°	Dry	23	80
Interlaminar Shear	Strength	MPa	EN 2563	0°	Dry	80	55
In-Plane Shear	Strength	MPa	EN 6031	±45°	Dry	23	120
In-Plane Shear	Modulus	GPa	En 6031	±45°	Dry	23	3.2

Figure 11: Mechanical properties of the TR50/M90 composite material

Nevertheless, this material has been recently developed and very few articles can be found in the literature considering this composite system. For this reason, considering the rick associated to the use of a recently developed composite material, it has been decided to not consider this material.

2.5. FOURTH ALTERNATIVE CANDIDATE: IM2C/M79 MATERIAL

Finally, Pipistrel proposed to consider the IM2C/M79, which is widely used for marine applications [17]. For instance, this material has been used for the manufacturing of composite foils in racing boats, as reported in Figure 12.

Figure 12: Use of IM2C/M79 for the manufacturing of foils in "Macif 100" racing boat

This material presents rather high mechanical properties (especially for longitudinal compression [17]) and interesting out-of-plane strengths [18], as reported in Figure 13, which are critical mechanical properties in order to design a composite foil. This material has been widely studied at IRDL (a research laboratory located at Brest in France) in order to design competitive racing boats. V. Keryvin, full professor at IRDL, has been contacted in order to share the available test results (tensile test on $[\pm45]_s$ and $[0_n]_s$ laminates subjected to tensile or compressive loadings, or four point bending test on L-angle specimens). He agreed to mutualize the available test results on this material. Since test results to determine the classical mechanical properties are available, the choice of such a composite material seems relevant.

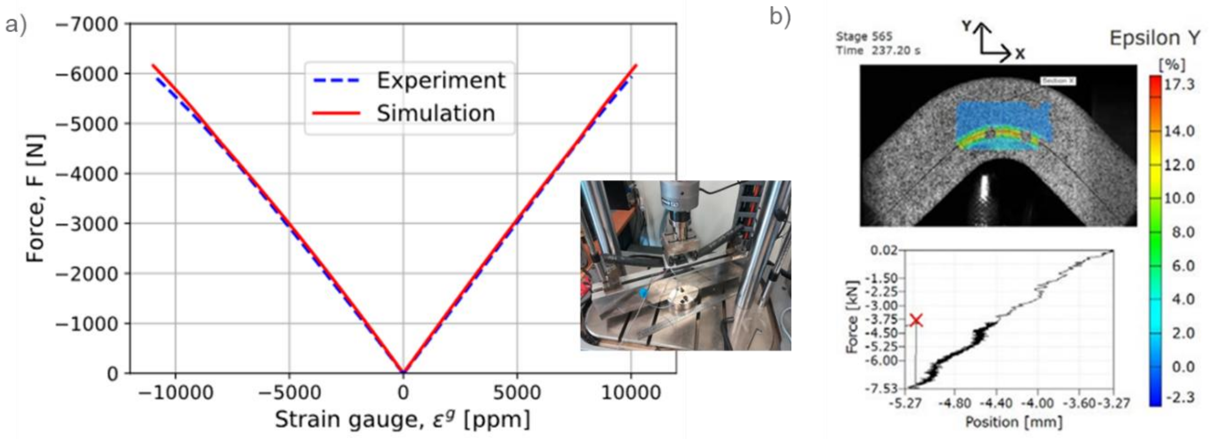

Figure 13: a) Bending tests to measure tensile and compressive behaviour in the fibre direction [17] and b) four point bending tests on L-angle specimens [18]

The manufacturing of this composite material is quite flexible because there is no need of additional pressure (except that of the vacuum bag) and the maximal curing temperature is about 80°C, which is consistent with the recommendations associated to the battery cells introduction. Moreover, this material is suitable to manufacture thick composite material because of a low exothermicity.

This material has also been considered for wind turbine applications [19–21], as illustrated in Figure 14.

Figure 14: Use of IM2C/M21 to manufacture large and thick wind blades [20]

Therefore, the fatigue lifetime has also been evaluated experimentally considering Wöhler curves and infrared thermography [22,23], as reported in Figure 15. The fatigue lifetime is rather high and interesting for wind turbine applications. These fatigue tests allow us to obtain an estimation of the fatigue lifetime of such a composite material compared to other classical material found in aeronautics, and it quite promising.

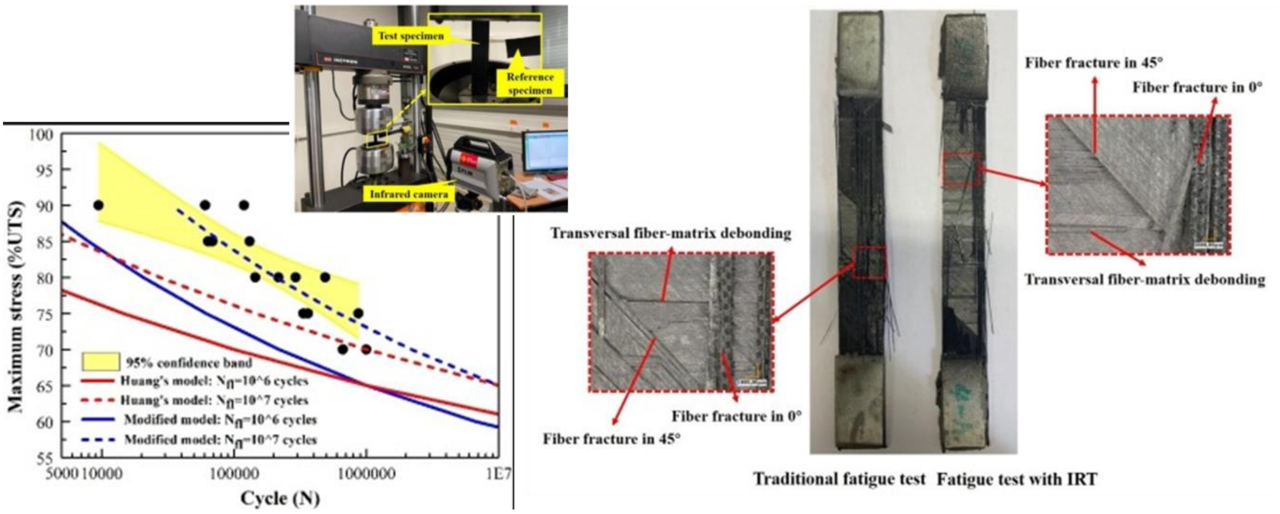

Figure 15: Fatigue lifetime estimation for IM2C/M21 with IR thermography [23]

Moreover, this material is considered in another research project lead by Pipistrel, and it has been estimated relevant to mutualize the experimental data. It must be considered that Hexcel does not guarantee that the composition of the composite material will not be modified in the next future. Nevertheless, the IM2C/M79 material has not been modified since the last 10 years (information provided by IRDL) and it could be reasonable to assume that the material will remain the same during the next years.

2.6. CHOOSEN COMPOSITE MATERIAL AND ASSOCIATED TEST CAMPAIGN

Considering the interesting technically and economically trade-off, and considering the synergy with other projects led by Pipistrel, the **IM2C/M79 composite material** has been chosen with an intermediate weight areal for unidirectional ply.

The material supplier *Composite Distribution* has been contacted by ONERA to obtain 200m^2 of the material with a delay of delivery about 6 weeks and a cost around 65 €/m^2, including the transport with a controlled temperature fixed at -18°C. Moreover, IRDL laboratory, who has already worked on this material, has been contacted to share the experimental data and thus limited the characterization test campaign.

Initially, the **test campaign planned in the DoA** considered the following types of tests:

- 36 quasi-static tests both on solid and sandwich composite materials to characterize material properties
- 51 impact tests on solid and sandwich composite plates with different energy levels and stacking sequences
- 8 fatigue tests to obtain a rough estimation of the fatigue lifetime of composite coupons with embedded battery cells

Because the composite material has been changed, it is necessary to adapt the test campaign to characterize the new composite material. It was decided to consider the same budget for the **new test campaign**, considering the following tests:

- ~50 quasi-static tests both on solid and sandwich composite materials to characterize material properties

- 17 impact tests on solid and sandwich composite plates with different energy levels
- 0 fatigue test (tests removed)

It was decided to remove the fatigue tests and reduce the number of impact tests because they are not mandatory in order to prepare the tests on the demonstrators, which is a winglet defined in WP5, planned at the end of the project. Among the 50 quasi-static tests, in-plane test on laminates ($[0_n]_s$, $[90_n]_s$, $[\pm 45_n]_s$ and $[45/0/-45/90]_{ns}$ laminates have been designed to estimate the intra-ply mechanical properties, delamination propagation test are also planned (DCB, ENF and MMB) with different mode mixings on $[0]_{ns}$ laminate, and finally adhesive tests are currently designed to estimate bounding between the composite material and the battery cells, which is a critical quantity in the project, as demonstrated by CIRA in WP3.

3. CONCLUSIONS

This report provides the details about the choice of the composite material which will be considered in the MATISSE project to (a) preserve the electrical performances of the battery cells developed by AIT, (b) enable an easy introduction of embedded SHM controls within the battery cells and within the composite material, and (c) ensure the manufacturing constraints imposed by Pipistrel (out-of-autoclave process) in order to obtain a technically and economically solution to produce the final demonstrator of the project.

It has been demonstrated, at the beginning of the project MATISSE, that the material considered in SOLIFLY project, named AS4/8552, despite many advantages, is not suitable considering an out-of-autoclave process. Therefore, it has been decided to change the initially planned material to another one. A literature survey has been performed mainly by ONERA and Pipistrel in order to determine potential candidates. Moreover, technical meetings with experts from *Hexcel* and *Composite Distribution*, major composite material providers, have been contacted to obtain additional information about the current composite market to be sure that the chosen solution could be applied in aeronautical industries in few years. Five carbon/epoxy composite materials have been widely studied and finally, the IM2C/M79 composite material made of unidirectional plies with an intermediate weight areal has been chosen and ordered.

Therefore, the characterization test campaign has been modified in order to characterize the missing mechanical properties of the composite itself and of the interface between the composite and the battery cells. The initially planned fatigue tests have been removed, and the number of impact tests have been reduced putting the focus on characterizing the mechanical properties required for the design and testing on the MATISSE winglet demonstrator.

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