New materials for fire protection in cabin environment – Test results second batch

I. Roese-Koerner (DLR), F. Martaus (VZLU), G. Mirra (LEONARDO), J. Bachmann (DLR), M. Liebisch (DLR)

Short abstract: Future Sky Safety is a Joint Research Programme (JRP) on Safety, initiated by EREA, the association of European Research Establishments in Aeronautics. The Programme contains two streams of activities: 1) coordination of the safety research programmes of the EREA institutes and 2) collaborative research projects on European safety priorities.

This deliverable is produced by the Project P7 “Mitigating the risk of fire, smoke and fumes”. The main objective is to present the results of the second batch tests on new materials for fire protection in the cabin environment.

Programme Manager	Michel Piers, NLR
Operations Manager	Lennaert Speijker, NLR
Project Manager (P7)	Eric Deletombe, ONERA
Grant Agreement No.	640597
Document Identification	D7.8
Status	Approved
Version	2.0
Classification	Public

This page is intentionally left blank

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

Contributing partners

Company	Name
DLR	I. Roese-Koerner, J. Bachmann, M. Liebisch
LEONARDO	G. Mirra
VZLU	F. Martaus

Document Change Log

Version	Issue Date	Remarks
1.0	01-01-2018	First formal release
2.0	27-02-2018	Second formal release

Approval status

Prepared by: *(name)*	**Company**	**Role**	**Date**
Jens Bachmann	DLR	Author	20-12-2017
Checked by: *(name)*	**Company**	**Role**	**Date**
Julien Berthe	ONERA	Quality assurance	21-12-2017
Approved by: *(name)*	**Company**	**Role**	**Date**
Eric Deletombe	ONERA	Project Manager (P7)	01-01-2018
Lennaert Speijker	NLR	Operations Manager	27-02-2018

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

Acronyms

Acronym	Definition
AITM	Airbus Industries Test Method
ASTM	American Society for Testing and Materials
CFRP	Carbon fibre reinforced plastic
CD	Cross Direction
CTE	Coefficient of thermal expansion
DLR	Deutschen Zentrums für Luft- und Raumfahrt
DMA	Dynamic mechanical analysis
DSC	Differential Scanning Calorimetry
GF	Glass Fibre
GP / GPL	Geopolymer
Gr	Gradient
F	Flammability / Flame Propagation
FAA	Federal Aviation Administration
FML	Fibre metal laminate
FRP	Fibre reinforced plastic
FST	Fire smoke toxicity
GFRP	Glass fibre reinforced plastic
HR	Heat release
HRR	Heat release rate
L	Linum usitatissimum (Flax), Layer
MD	Machine Direction
OSU	Ohio State University
PF	Phenolic Resin (phenol formaldehyde)
rCF	Recycled Carbon Fibre
S	Smoke
SD	Smoke Density
RTM	Resin transfer moulding
T	Toxicity

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

TGA	Thermogravimetric analysis
TMA	Thermomechanical analysis
vCF	Virgin Carbon Fibre
VZLU	Czech Aerospace Research Centre

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

EXECUTIVE SUMMARY

Problem Area

Many studies on the current flights show that about 50% of the fatalities in case of aircraft accidents are linked to situations where fire is involved. Hundreds of fatalities could be saved per year if fire effects on the primary structure or in the cabin environment were mitigated. The development of larger, more electric and more lightweight aircraft (with an increase use of Carbon Fiber Reinforced Plastic (CFRP) composite parts) raises several safety questions with respect to unknown behaviours of the materials and structures when exposed to fire. But the scope of this problem is large, embracing a variety of problems and solutions: the use of fireproof and less toxic materials, the early detection of fire, the simulation of passengers' evacuation, etc.

Future Sky Safety Project P7 "Mitigating the risks of fire, smoke and fumes" will focus on effects of fire on new materials with improved fire properties (production of heat, toxic fumes and smokes), and on the effect of fire on mechanical behaviour that can endanger the passengers' life. The scope of the works will cover both primary structures materials (e.g. epoxy resin, carbon fibre reinforced polymers) and cabin materials (e.g. phenolic polymers, glass fibre reinforced plastics). The objective of WP7.2 is to develop and utilize novel and innovation material solutions with high potential for mitigating risks of fire, smoke and fumes in the cabin environment. To achieve this aim, proposed highly resistant materials will be tested according to prescribed test plan, which will allow addressing their mechanical properties with respect to fire exposure. The scope and magnitude of proposed test plan respect industrial safety requirements and usage of state-of-the art simulation tools.

Description of Work

Deliverable D7.2 provided the requirements and specifications of the tests. The scope and magnitude of the test plan defined for the experiments and the data content respect industrial safety requirements and usage of state-of-the art simulation tools. D7.5 was dedicated to the test results from the first batch of tests. D7.8 is a summary of results from the second batch of tests in WP7.2 of the FSS project

Results & Conclusions

GEOPOLYMERS

The second batch of geopolymers was focused mainly on sandwich structures and included following tests:

Fire Effluents and Smoke Optical Density of Carbon Fiber Geopolymer Composites.

Parameters of thermal decomposition effluents and smoke optical density of carbon fiber/geopolymer and referential glass fiber/phenolic composites were examined. Geopolymer resin GPL30 (VZLU) was applied. GURIT PHG 600-68-37 T2 glass/phenolic prepreg was used as referential material. The tests were

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

carried out in accordance with standards *EN ISO 5659-2: 2013 Plastics – Smoke generation – Part 2 Determination of optical density by a single-chamber test* and *Fire Technical Institute Guideline No. 01-09, procedure B*. Heat flux density of 25 kW/m^2 was applied on all test specimens.
From the point of view of criteria under review, carbon fiber reinforced geopolymer gives better results in comparison with referential glass/phenol in all evaluated parameters.

Flame Penetration Test per CS 25 Appendix F Part III.

Tested sandwich panels featured both foam and honeycomb cores. Panels skins were made of carbon fiber reinforced geopolymer composite. As the referential test specimens, GURIT PHG 600-68-37 (style 7781) glass/phenolic co-cured sandwich panels were employed. No fire penetration was indicated both on carbon/geopolymer and referential glass/phenolic panels, regardless of the sandwich core material. Temperature measured 102 mm (4") above the upper surface was exceeded in case of glass/phenolic panel. Referential glass/phenolic panels typically have ruptured during the test resulting in smoke effluents escape from burned core. No mechanical damage or ruptures and strong "pillow effect" were indicated on carbon/geopolymer panels.

Impact Tests of Carbon/Geopolymer and Carbon/Geopolymer/Phenol Sandwich Panels

Impact tests of sandwich panels were carried out. The panels were constructed of Airex R82.60 structural foam core and:

a) carbon fiber / geopolymer skins
b) carbon fiber / geopolymer - carbon fiber / phenol hybrid skins

The tests were carried out in accordance with *ASTM D 7136/D 7136M – 07 standard (Standard Test Method for Measuring the Damage Resistance of a Fiber-Reinforced Polymer Matrix Composite to a Drop-Weight Impact Event)*. The group of samples was exposed in 70^{o}C / 85% RH hot-wet conditions for 2 weeks. Visible Impact Damage (VID) and Barely Visible Impact Damage (BVID) were evaluated. In the VID mode both TYPE 1 (geopolymer only skins) and TYPE 2 (hybrid skins) specimens surprisingly show better impact resistance after hot - wet exposition than not-exposed specimens. Possibly it is an attribute of additional post-curing of geopolymer matrix during the exposition in the climatic chamber. In the BVID mode TYPE 1 specimens showed practically no sensitivity to hot - wet exposition. TYPE 2 specimens exhibit drop of impact resistance as expected. Generally, TYPE 2 (hybrid skins) showed better resistance against the impact as presumed.

Comparison of TYPE 1 and TYPE 2 specimens to referential specimens made of carbon/epoxy showed worse impact resistance of both geopolymers and hybrids.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

Drum Peel Tests of Carbon Geopolymer Sandwich Panels.

Peel strengths of sandwich panels constructed of carbon fiber reinforced geopolymer skins and honeycomb resp. foam cores were evaluated. As referential group of specimens, GURIT PHG 600-68-37 (style 7781) glass/phenolic prepreg based, co-cured sandwiches were employed. Various skins/adhesive/cores material combinations were examined. The tests were carried out in accordance with *ASTM D1781 - 98 standard (Standard Test Method for Climbing Drum Peel for Adhesives)*, in normal conditions, with no previous environmental exposition. In the group of foam core specimens the best results showed GPL30 laminating resin bonded specimens, closely followed by PH 600 prepreg bonded samples. In the group of honeycomb core specimens the best results showed Resbond® 989 bonded specimens, followed by PH 600 prepreg bonded samples. Generally, foam core specimens provided better test results.

Compression Test of the Geopolymer Foam

Development of composition and processing of inorganic (geopolymer) based structural foam was carried out. Compression tests of the foam per *ASTM D1621* standard were performed. Development of more lightweight foam is currently in the progress. Test specimens of specific weight of ca 200 kg/m^3 are being tested.

ECO-FIBRES

The use of bio-fibres to substitute glass fibres in interior composite materials for aviation (passenger and cargo compartment) could be beneficial for the environmental impact. The same is expected for the application of valuable recycled carbon fibres from cutting waste or end-of-life products via pyrolysis process. Flax fabric (plain weave) and a nonwoven from rCF are used as reinforcement in the second batch of tests. As the geopolymer (GPL) matrix used by partner VZLU shows very good fire properties, it was the aim of the second test batch to combine the ecological beneficial fibres (flax and rCF) with the geopolymer matrix. Fire tests according to FAR for cargo compartment (F, ST, HR) and basic flexural tests have been conducted to show the potential advantages and challenges of these material combinations. The results show very promising FST and HR results for the rCF nonwoven with Geopolymer matrix with advantages compared to the state-of-the-art glass fibre phenolic resin (GF-PF) combination, e.g. all toxic gases could be reduced. Flammable flax fibres in combination with geopolymer matrix show good flammability values and toxic gases below the limit. Depending on the amount of flax fibres used in the composites, the heat release limit of has been exceeded. Therefore further investigations to add a flame retardant are needed for the application of bio-fibres in aviation interior linings. A hybrid combination of out layers from rCF and inner layers of flax show promising FST + HR results in the range of the GF-PF reference. The mechanical properties need to be improved by a better fibre-matrix adhesion.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

FIBRE METAL LAMINATES

The second test batch consists of further tests for characterization of FML material properties. The tests contained DMA measurements as well as the determination of temperature dependent shear and compression properties. All Tests were conducted for FML and the used unidirectional prepreg material that was used for FML processing. The results show dropping properties for strength and shear properties with moderate amount until 150°C and higher degradation above 150°C. The compression modulus in fibre direction is almost not influenced until 150°C.

Moreover, the second batch includes fire tests with simultaneous mechanical loading. To this, a test stand was developed. The test stand allows axial compression loading of curved specimens that are potted within concrete-filled moulds. The moulds are mounted to a press. Within the test, the specimens are preloaded by 50MPa axial compression load. The test stand construction includes additional insulation and allows fire loading to the specimen while the compressive force is still loaded. Multiple tests have been conducted on FML specimens showing the pillow effect that works as insulation to the rear laminate plies. Additional to the burn-through resistance of FMLs, the structural integrity was investigated with respect to such a fire scenario. The collapse of the structure was investigated to be after sideway cracking of the developed pillows. As a consequence to this, temperature rises at the rear plies causing locally decreasing mechanical properties resulting in structural collapse.

Applicability

The tests have been executed on the basis of the requirements and tests defined in deliverable D7.2. Aim of the tests:

- Verification of the compliance with the certification requirements;
- Measure of the material characteristics.

The applicability is dual:

- Evaluate the material capacity to withstand at high temperature/fire condition;
- Use the material characteristics measured as input data for activity of experimental/numerical correlation of the simulation model.

This page is intentionally left blank

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

TABLE OF CONTENTS

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

LIST OF FIGURES

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

LIST OF TABLES

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

1 INTRODUCTION

1.1. The Programme

The European Commission (EC) Flight Path 2050 vision aims to achieve the highest levels of safety to ensure that passengers and freight as well as the air transport system and its infrastructure are protected. However, trends in safety performance over the last decade indicate that the ACARE Vision 2020 safety goal of an 80% reduction of the accident rate is not being achieved. A stronger focus on safety is required. There is a need to start a Joint Research Initiative (JRI) for Aviation (Future Sky) with a Joint Research Programme (JRP) on Safety, and also for coordination of Safety Research conducted under the Institutional Programs of the European research establishments. The JRP on Safety (Future Sky Safety), established under coordination of the Association of European Research Establishments in Aeronautics (EREA), is built on the relevant European safety priorities as brought forward in Flightpath 2050 and the European Aviation Safety Plan. The program is structured around four main themes with each theme consisting of a small set of projects. Theme 1 (New solutions for today's accidents) aims for breakthrough research with the purpose of enabling direct, specific, significant risk reduction for the two main Accident Categories. Theme 2 (Strengthening the capability to manage risk) conducts research on processes and technologies to enable the aviation system actors to achieve near-total control over the safety risk in the air transport system. Theme 3 (Building ultra-resilient systems and operators) conducts research on the improvement of Organizations, Systems and the Human Operator with the specific aim to improve safety performance under unanticipated circumstances. Theme 4 (Building ultra-resilient vehicles), aims at reducing the effect of external hazards on the aerial vehicle integrity, as well as improving the safety of the cabin environment. In all, Phase 1 of the Programme will address five important safety priorities. The Project P7 "Mitigation the risk of fire, smoke and fumes", contributes to Theme 4 "Building ultra-resilient vehicles" of the Future Sky Safety Programme.

1.2. Project context

The first objective of the P7 project is to increase safety - meaning here reduce the number of fatalities - with respect to fire related issues (in-flight or post-crash). First, many studies on the current flights show that about 50% of the fatalities in case of aircraft accidents are linked to situations where fire is involved. Hundreds of fatalities could be saved per year if fire effects on the primary structure or in the cabin environment were mitigated. Second, the development of larger, more electric and more lightweight aircraft (with an increased use of CFRP composite parts in A/C design, such as fuselage panels, engine carters, engine exhausts, etc) raises several safety questions with respect to unknown behaviours of the materials and structures when exposed to fire. But the scope of this problem is large, embracing a variety of problems and solutions: the use of fireproof and less toxic materials, the early detection of fire, the simulation of passengers' evacuation, etc. And few researches have been funded yet by the EU commission on this subject. Compared to the previous 7th PCRD funded project "AircraftFire", on which the JRI one is nevertheless built on, it was decided to address the fire issue in the JRI Safety research

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

programme as part of Theme 4: “Building the Ultra-resilient Vehicles”. It means that the research work will be focused on material and structural questions, and will aim at mitigating fire related safety risks when/by introducing new generation of materials in future aircraft design (incl. possible eco-friendly ones). Considering this focus, it must be noticed that very few test results are available today to the research community, because of obvious costs (test facilities, destructive tests, specimens and sensors) and industry confidentiality reasons. A large part of the project will be dedicated to develop and share experimental testing facilities and test results, with a clear partnership added value between EU Research Establishments, Academia and Industry being reached.

Then, there are also concerns about the safety impact on on-board air quality, mainly related to such innovations. For example, aircraft crew has reported health problems that prevent them from working appropriately and which they consider to be due to air quality in the cockpit and cabin of pressurised aircraft. Multiple investigations have been carried out on hypothetical air contamination by oil ingredients and on the potential impact of such contamination on occupants’ health, both in short term and in long term. The more general question of any possible kinds of impact on on-board air quality then raised, that can be due for instance to the introduction of new materials in the design that could react with more and more electrical heating systems, fuel systems, or in case of fire which can then be linked to the previously mentioned first objective.

The project will then address on the one hand effects of fire on materials (production of heat, toxic fumes and smokes), and on the other hand effects of fire on structures (burn-through, strength) that can endanger the passengers’ life directly (exposure) or indirectly (evacuation). The scope of the works cover both primary structures materials (e.g. epoxy resin, carbon fibre reinforced polymers) and cabin materials (e.g. phenolic polymers, glass fibre reinforced plastics). Last, the P7 project has been split into three work packages according to the expected impacts that are claimed for this 3 years research work:

- WP7.1 - aims at improving the knowledge about effects of fire on materials and structures. This sub-work package would mainly concern standard epoxy resins and carbon fibres reinforced polymer materials (primary structures),
- WP7.2 - the second one aims at proposing improved materials solutions, mainly to mitigate fire, smoke and fumes. This second work package would concern new materials (primary structures and cabin), the properties of which will be compared to standard ones,
- WP7.3 - aims at analysing possible effects on the on-board air quality that the introduction of such new materials in the aircraft structure and cabin could have.

1.3. Work package context

The objective of WP7.2 is to develop and utilize novel and innovative material solutions with high potential for mitigating risks of fire, smoke and fumes in the cabin environment. To achieve this aim, proposed highly resistant materials are being tested according to prescribed test plan, which should allow to address their mechanical properties with respect to fire exposure. The scope and magnitude of proposed test plan respect industrial safety requirements and usage of state-of-the art simulation tools.

Future Sky Safety has received funding from the EU’s Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

The objective of this investigation concerning fibre metal laminates is the development of FMLs with improved fire properties for the substitution of cabin and structural aircraft materials. This material combination offers the opportunity of a reduced smoke density production with a lower toxic gas content combined with improved mechanical properties during fire.

The objective of hybrid non-woven from natural fibres and (recycled) man-made fibres is to substitute classic cabin materials (glass fibre fabric) with more ecological friendly materials. The use of recycled carbon fibres will enhance the mechanical properties and also improve the fire properties to mitigate the risk of fire and fumes in the cabin environment.

The objective of utilization of geopolymers matrices reinforced by carbon fibers is to test innovative material systems providing limited smoke and toxic gas content with sufficient mechanical properties during fire exposure for passenger and cargo linings. Versatility of geopolymers matrices allows their exploitation both on laminate and sandwich structures, where e.g. foam could provide significant impact on mitigating the risk of fire and fumes in the cabin environment.

1.4. Research objectives

The main objective of this study is the presentation of the results of the second test batch based on studied materials to improve material solutions to mitigate and protect from fire, smoke and fumes in the cabin environment (plus toxicity).

1.5. Approach

The combination of metal layers and CFRP (fibre metal laminates) can lead to better FST properties. In this project the effect of different thicknesses, numbers and places of the metal layers should be investigated. For this, FMLs with different lay-ups have been manufactured and tested with a special focus on FST properties.

Another approach is to use semi-finished products from natural and recycled carbon fibres in combination with flame resistant geopolymer matrix.

Exploitation of geopolymer matrices in combination with standard composite materials could pave way to acceptable overall mechanical properties of geopolymers material systems. Application of carbon/geopolymer, hybrid geopolymer / phenolic systems or geopolymer foam should be tested with respect of chosen manufacturing processes from coupon up to linings level.

1.6. Structure of the document

Chapter 1 covers an introduction summarizing:

- the programme content
- the P7 research objectives and approach

In Chapter 2 the proposed innovative materials solutions are defined.

Chapter 3 provides an overview of test results of the first batch together with description of manufacturing processes needed for production of testing panel and coupons..

Conclusions and Recommendations are discussed in Chapter 4.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

2 MATERIALS

The following three subchapters will give detailed information about the materials that will be used and reveal their potential regarding fire protection.

2.1. Geopolymers

Geopolymers (GP) are amorphous inorganic aluminosilicate materials that combine low temperature, polymer-like processing with high temperature stability (Fig. 1). This combination of properties makes geopolymers an interesting alternative to existing organic polymeric and ceramic matrix materials and offers a high potential for the development of cost-efficient, ceramic matrix - like composites for applications in the mid to high temperature (up to ca 1200°C) range.

Geopolymers feature some unique characteristics, e.g. high temperature stability and fire resistibility, achieved by low temperature processing. GP are also characterized by generation of almost no toxic fumes and smokes, low thermal conductivity, low cost and simple preparation.

On the other hand, strength parameters of GP composites are lower than of organic ones and GP matrix features brittle properties.

Geopolymer matrix has potential to be utilized in heat and fire resistant composites in aerospace structures and have ambition to fulfil and even exceed present FST requirements.

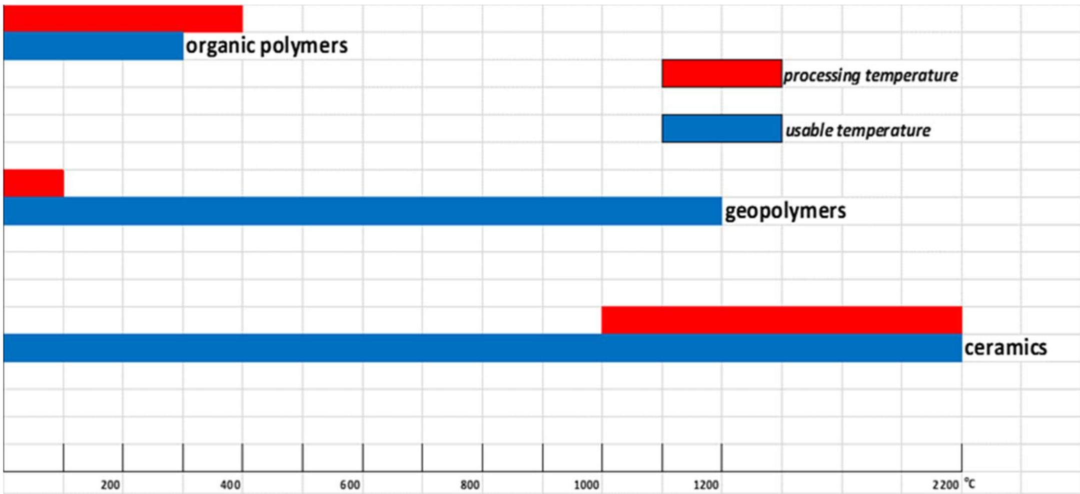

Figure 1: Processing and usable temperatures „windows" of composite matrixes

As the base matrix material for scheduled test program, GPL30 geopolymer system developed by VZLÚ, a.s., is established. GPL30 is a low viscosity geopolymer matrix system optimised for use in thin walled composite shell structures. It is appropriate for most of basic processing methods. GPL30 matrix system doesn´t have any commercial equivalent at the present, regarding MEYEB and DAVYA resin systems delivered by Institut Géopolymère are not available on the market anymore. Composition and preparation of GPL30 geopolymer resin is stated in a separate VZLÚ report.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

As a reinforcement phase, carbon fiber in form of woven fabrics, NC fabrics, unidirectional tapes and recycled non-wovens are established due to their mechanical and physical properties suitable for intended application. Natural fibers (flax) in form of woven fabric are also subject of the testing. If glass fibers are utilized, strong alkaline character of GP matrix shall be considered. To avoid corrosion of standard glass fibers, special corrosion resistant glass, e.g. OCV Advantex® fibers shall be employed.

2.2. Eco-fibres

The use of bio-fibres to substitute glass fibres in interior composite materials for aviation (passenger and cargo compartment) could be beneficial for the environmental impact. The same is expected for the application of valuable recycled carbon fibres from cutting waste or end-of-life products via pyrolysis process. Both, flax fibres and recycled carbon fibres (rCF), have been assessed in the first test batch of WP7.2 in form of hybrid nonwoven manufactured in the DLR laboratory. Natural fibres contain mainly cellulose with smaller amounts of hemicellulose, pectin and lignin. Fire properties are a main drawback when using natural fibres as substitution for glass fibres. Therefore a combination with a matrix system with intrinsically good fire properties would be beneficial to avoid the use of high amounts of flame retardants that reduce mechanical properties (matrix filler, fibre sizing) and increase the composite weight (coating).

Table 1: Properties from the flax fibre fabric data sheet [LINEO BL300 data-sheet, available online: www.lineo.eu/products]

Property	Unit	Standard	Value
COMPOSITION *Reinforcing fiber : Flax X Hemp □*	VOL %		100 ±
AREAL WEIGHT	g / m²	ISO 3801	318 ±
*AREAL VOLUME **	mm³ / mm²	Calculation	0.219 ±
THICKNESS	mm	ISO 5084	0.3 ±
WEAVE STYLE			Twill 2/2
YARNS / CM	(warp)	ISO 4602	12.1 ±
PICKS CM	(weft yarns)	ISO 4602	10.2 ±
WEIGHT DISTRIBUTION	%		weft : 44.3 ± warp : 45.7 ±
STANDARD WIDTH	cm	ISO 5025	103 ±

For the second test batch, the focus has been shifted towards commercially available flax fabric (plain weave, 318g/m², Table 1) and a nonwoven from rCF (Carbiso M, 100g/m², Table 2, Figure 2) supplied by the UK based recycling company ELG. As the geopolymer (GPL) matrix used by partner VZLU shows very good fire properties, it was the aim of the second test batch to combine the ecological beneficial fibres (flax and rCF) with the geopolymer matrix. Fire tests according to FAR for cargo compartment (F, ST, HR) and basic flexural tests will help to assess the potential advantages and challenges of these material combinations. For more detailed information about the geopolymer matrix used by VZLU, please visit chapter 2.1.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

Table 2: Properties from the recycled carbon fibre nonwoven [ELG Carbiso M data-sheet, available online: http://www.elgcf.com/assets/datasheets/Carbiso-M.pdf]

Typical properties	Units	Values
Fibre length	mm	40 – 90
Areal weight	g/sqm	100 – 500
Fabric weight tolerance	%	±8
Roll length (500g/sqm)	m	30
Roll diameter (500g/sqm)	mm	1000
Roll width	mm	500 – 2700
Core ID	mm	152.4

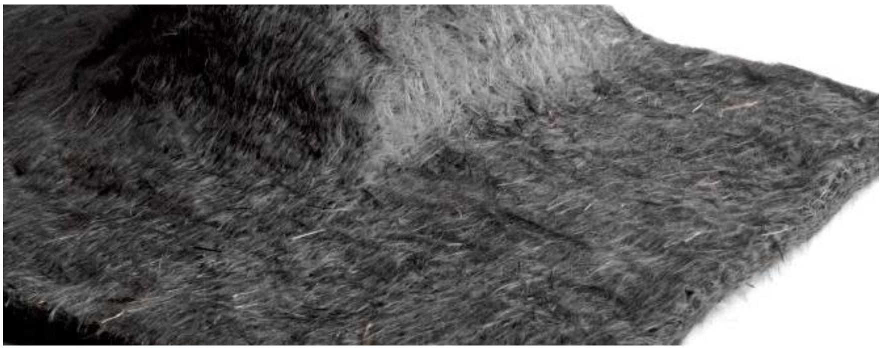

Figure 2: rCF nonwoven "Carbiso M" by ELG Carbon Fibre Ltd. [ELG Carbiso M data-sheet, available online at: http://www.elgcf.com/assets/datasheets/Carbiso-M.pdf]

2.3. Fibre Metal Laminates

Fibre metal laminates (FML) are hybrid materials which consist of several thin metal layers bonded with layers of composite material. Figure 1 shows the lay-up schematically.

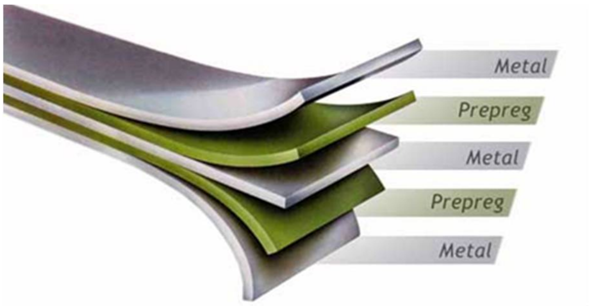

Figure 3: Lay-up of fibre metal laminates (schema) [http://unitedglassply.com]

A standard and for aerospace certified prepreg based on unidirectional carbon fibres with an epoxy resin matrix was and will be used in this project. This prepreg material exhibits an excellent reliability with respect to environmental influences and aggressive media combined with outstanding mechanical properties. The metal layers will be made of stainless steel type 1.4310 with various thicknesses. This

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

material is able to create a good bonding to the epoxy matrix and has mechanical properties, like elongation, that are compatible with carbon fibre prepreg layers.

In further investigations, it is found that the combination of metal layers and CFRP layers can lead to better FST properties. This effect is partly based on the different thermal coefficients of expansion of the materials. In case of fire, it is a source for delamination which forms a gap in the material. So doing, the underlaying structure is detached and protected for a certain amount of time. In addition, the metal layer can act as a barrier for smoke and fumes and thus restrict the combustion process.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

3 RESULTS OF SECOND TEST BATCH

The following chapter will give some general information about the production of fibre reinforced plastics, of fibre metal laminates as well as of eco-fibre materials. Furthermore, main test results are presented.

3.1. Geopolymers

3.1.1. Fire Effluents and Smoke Optical Density of Carbon Fiber Geopolymer Composite

Parameters of thermal decomposition effluents and smoke optical density of carbon fiber/geopolymer and referential glass fiber/phenolic composites were examined.

Geopolymer resin GPL30 in locked-in composition was applied. GURIT PHG 600-68-37 T2 glass/phenolic prepreg was used as referential material.

The tests were carried out in accordance with standards:

- EN ISO 5659-2: 2013 Plastics – Smoke generation – Part 2: Determination of optical density by a single-chamber test
- Fire Technical Institute Guideline No. 01-09, procedure B.

Heat flux density of 25 kW/m^2 was applied on all test specimens.

Detailed information and unabridged test protocols are stated in the VZLU report No.: R-6759/2017.

Acronyms

CIT4min	Conventional Index of Toxicity in the 4th minute
CIT8min	Conventional Index of Toxicity in the 8th minute
CO	Carbon monoxide
CO2	Carbon dioxide
Dc	Specific optical density of smoke after the measurement
Ds10	Specific optical density of smoke in the 10th minute
Ds4	Specific optical density of smoke in the 4th minute
Dsmax	Maximal specific optical density of smoke
FED30min	Fractional effective dose
VOF4	Accumulated value of specific optical density of smoke in the first 4 minutes

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

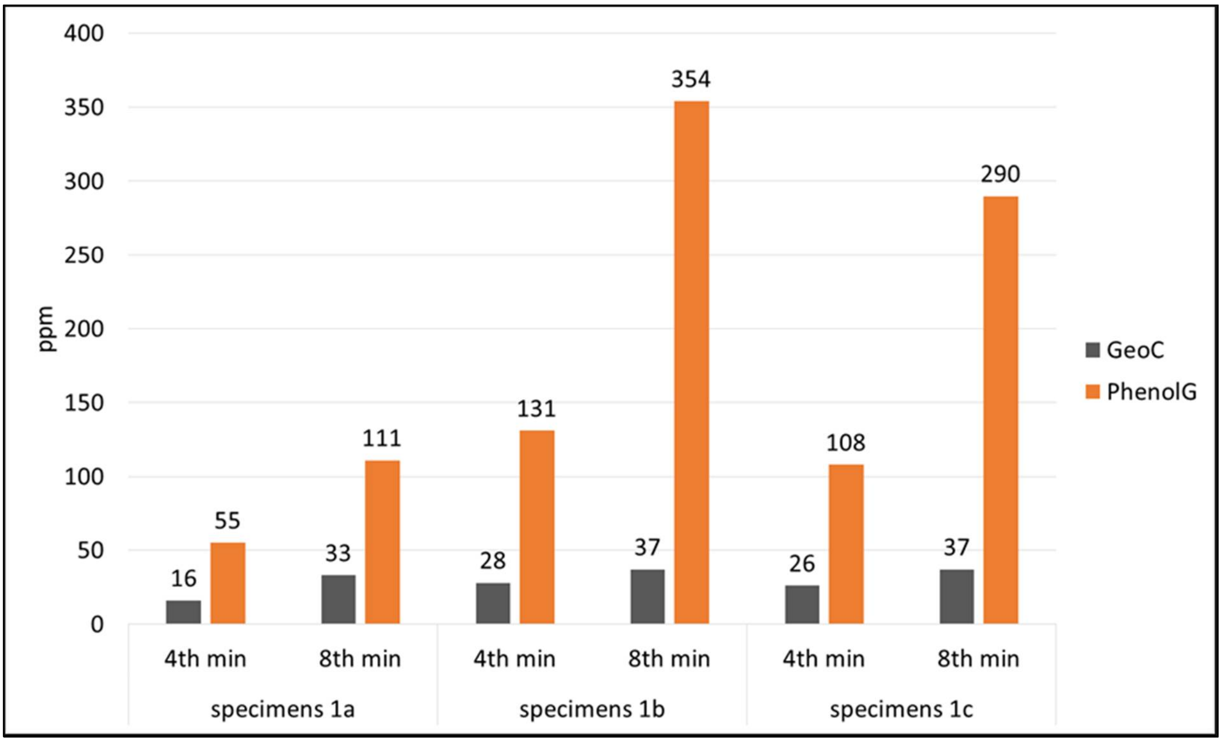

Figure 4: CO concentrations under test conditions

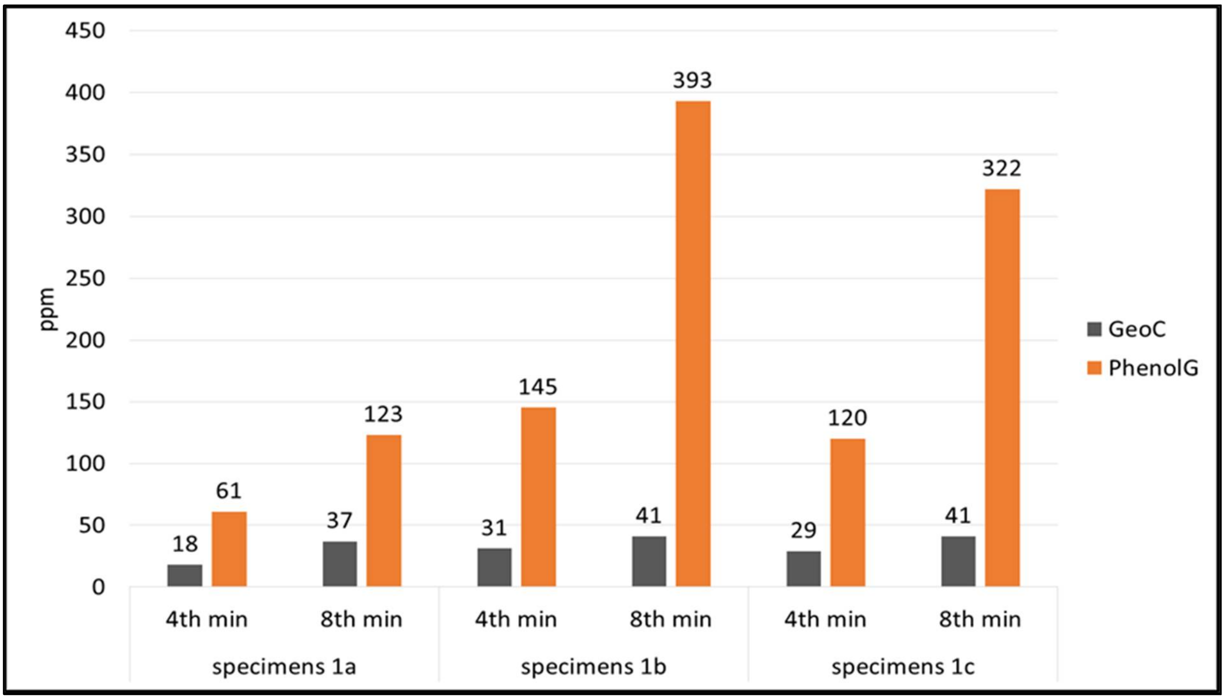

Figure 5: CO concentrations converted to standard pressure and temperature (101.325 kPa, 25°C)

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

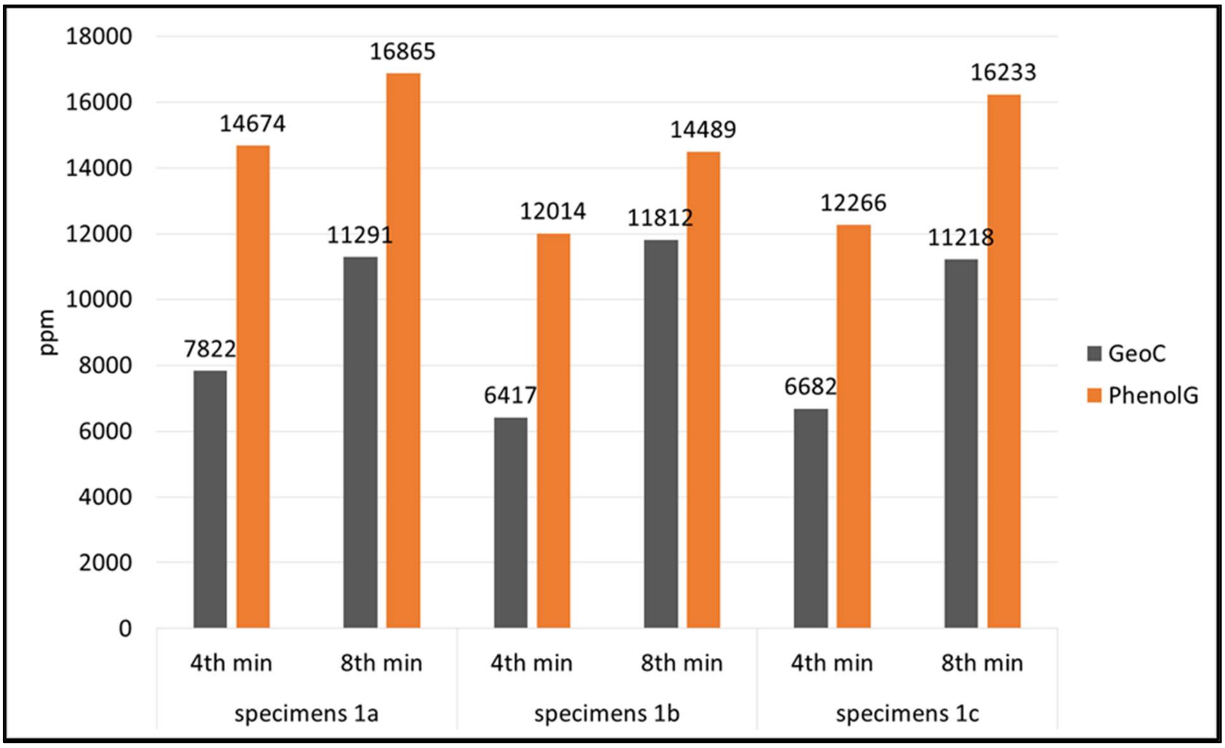

Figure 6: CO2 concentrations under test conditions

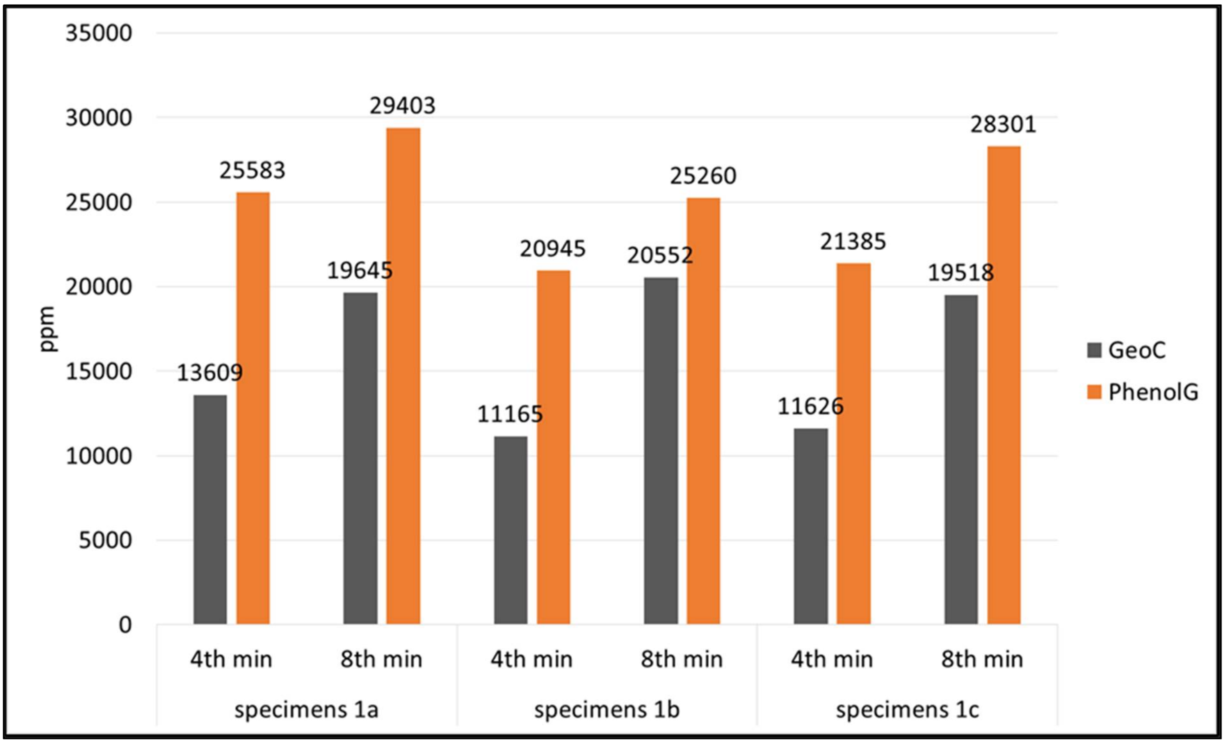

Figure 7: CO2 concentrations converted to standard pressure and temperature (101.325 kPa, 25 °C)

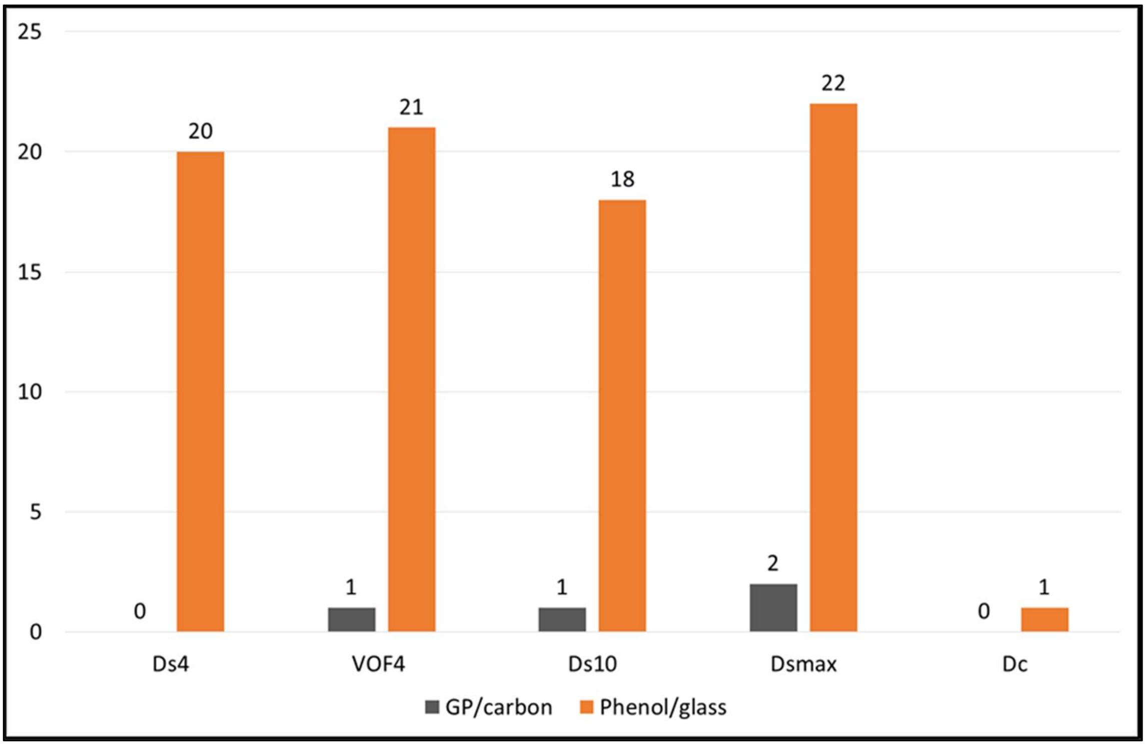

Figure 8: Smoke optical density

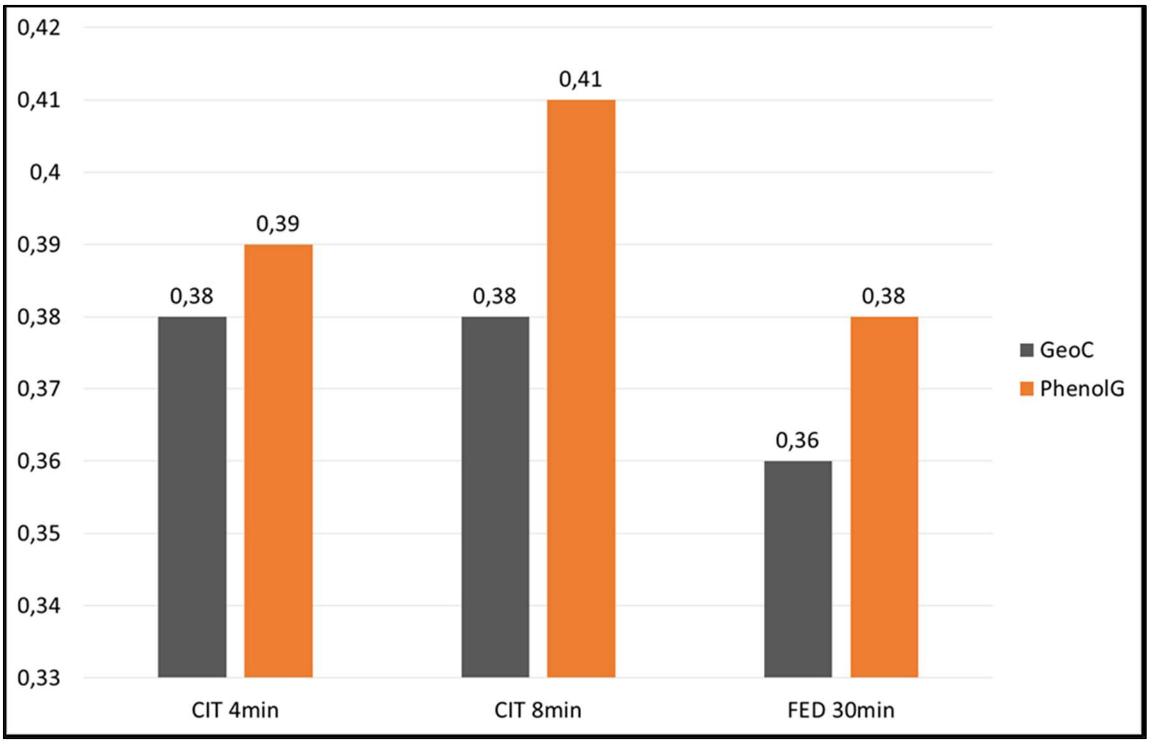

Figure 9: Conventional index of toxicity in the 4th minute (CIT4min), in the 8th minute (CIT8min) and total fractional effective dose (FED30min)

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

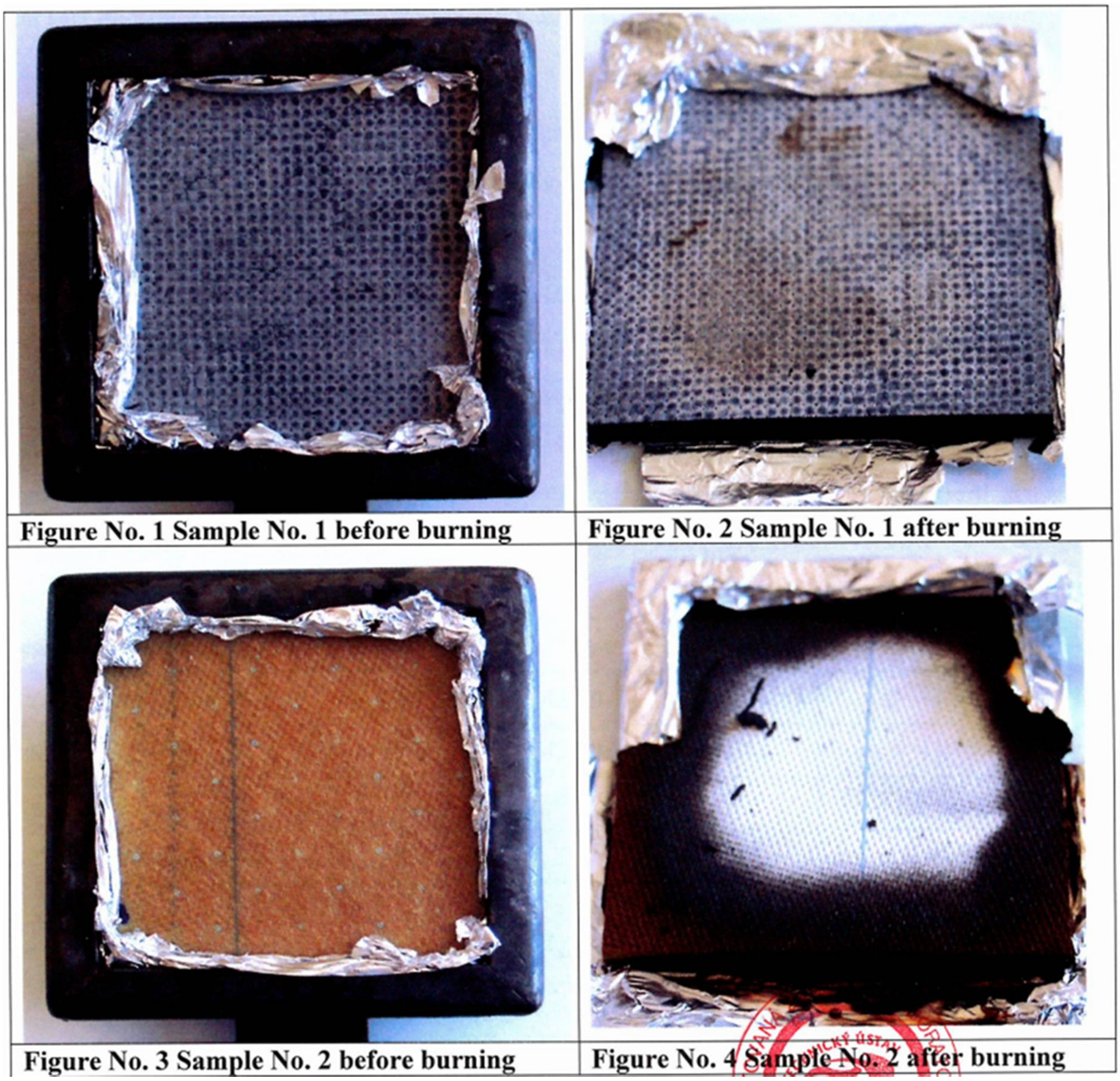

Figure 10: Materials samples before and after burning. Carbon / geopolymer above (Fig. No. 1, Fig. No. 2), glass / phenol below (Fig. No. 3, Fig. No. 4). White spot surrounded by soot on Figure No. 4 is a glass fabric where phenolic resin was completely burned off

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

3.1.2. Flame Penetration Test per CS 25 Appendix F Part III (Flame Penetration Resistance of Cargo Compartment Liners).

The sandwich panels featured both foam and honeycomb cores. Panels skins were made of carbon fiber reinforced geopolymer composite.

As the referential test specimens, GURIT PHG 600-68-37 (style 7781) glass/phenolic co-cured sandwich panels were employed.

Detailed information and unabridged test protocols are stated in the VZLU reports No.: U_MOT_0007 and U_MOT_0010.

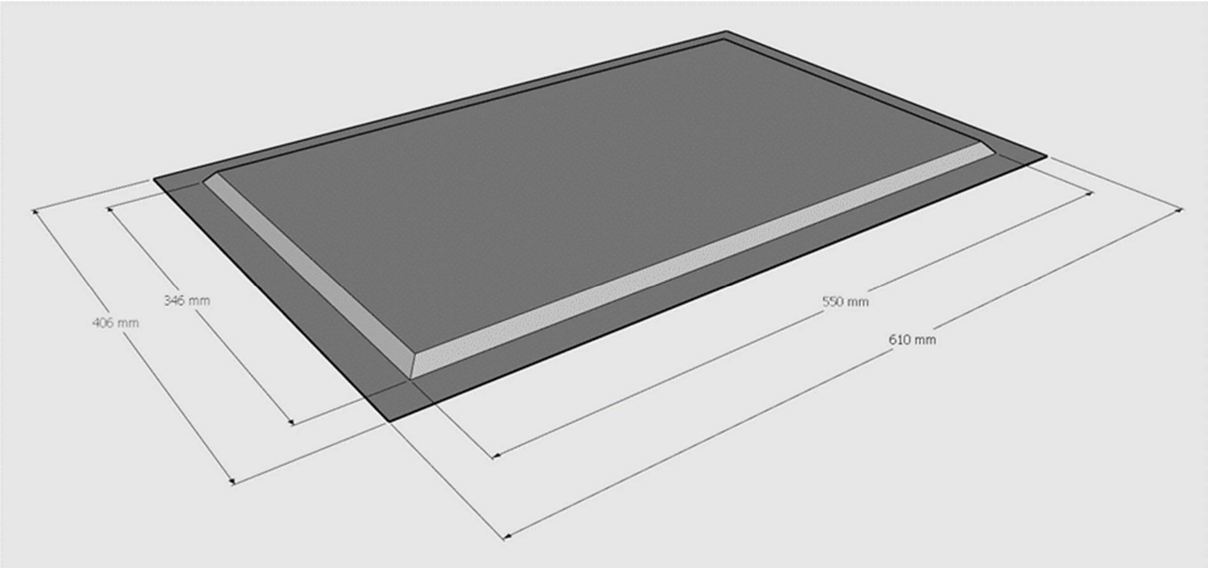

Figure 11: Sandwich test panel – geometrical scheme (core thickness 10 mm, core edges 45o chamfered)

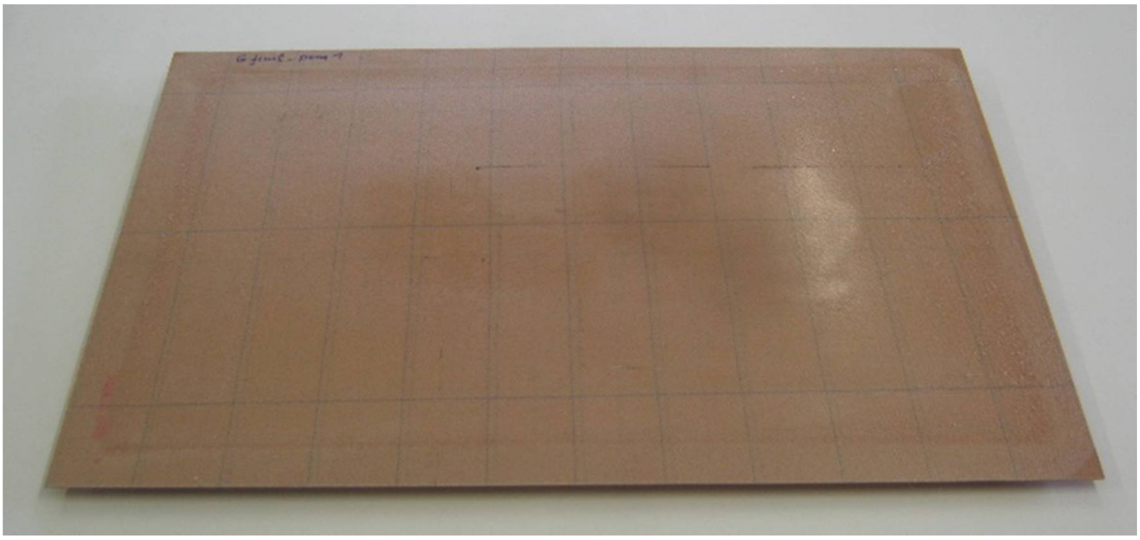

Figure 12: Exposed side of the referential glass/phenolic panel before the test

Figure 13: Exposed side of the referential glass/phenolic panel after the test. White spot is a glass fabric where phenolic resin was completely burned off. Typical rupture can be seen on the edge of the panel

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

Figure 14: Exposed side of carbon/geopolymer panel before the test

Figure 15: Exposed side of carbon/geopolymer panel after the test. Typical “pillow effect” is evident

Future Sky Safety has received funding from the EU’s Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

Figure 16: Split carbon/geopolymer panel after the test. No fire penetration or ruptures were registered.

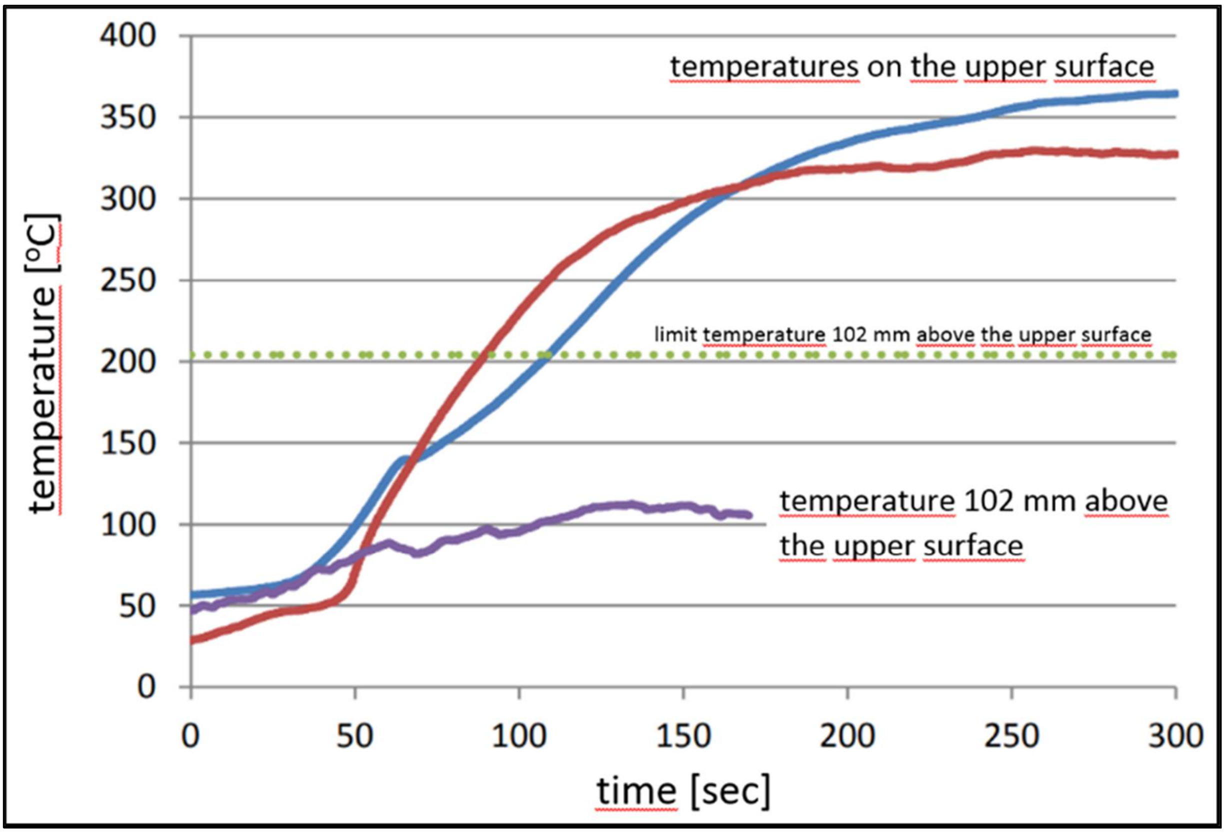

Figure 17: Carbon/geopolymer panel (honeycomb core): flame penetration test temperature record

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

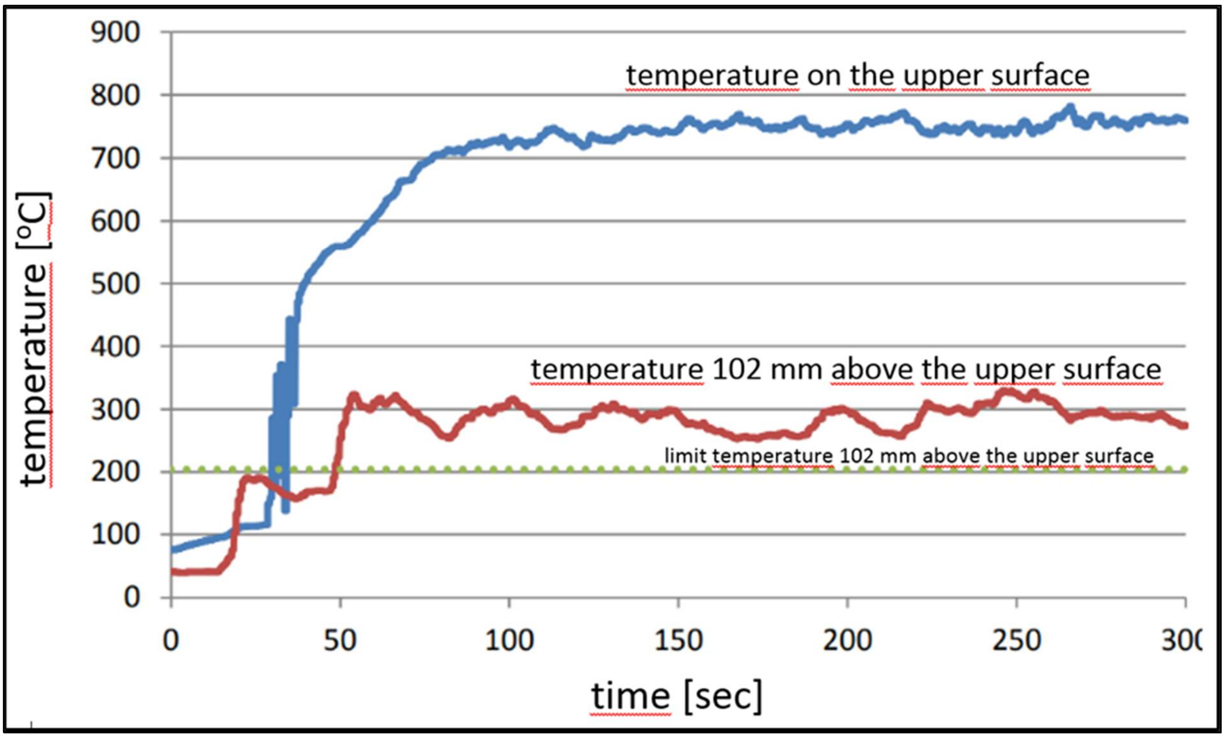

Figure 18: Referential glass/phenolic panel (honeycomb core): flame penetration test temperature record

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

3.1.3. Impact Tests of Carbon/Geopolymer and Carbon/Geopolymer/Phenol Sandwich Panels

Impact tests tests of sandwich panels were carried out. The panels were constructed of Airex R82.60 structural foam core and:

a) carbon fiber / geopolymer skins

b) carbon fiber / geopolymer - carbon fiber / phenol hybrid skins

The tests were carried out in accordance with ASTM D 7136/D 7136M – 07 standard (Standard Test Method for Measuring the Damage Resistance of a Fiber-Reinforced Polymer Matrix Composite to a Drop-Weight Impact Event).

The group of samples was exposed in 70oC / 85% RH hot-wet conditions for 2 weeks. The exposition process was carried out per EN 60068-2-78 standard.

Detailed information and unabridged test results are stated in the VZLU report No.: R-6722/2017.

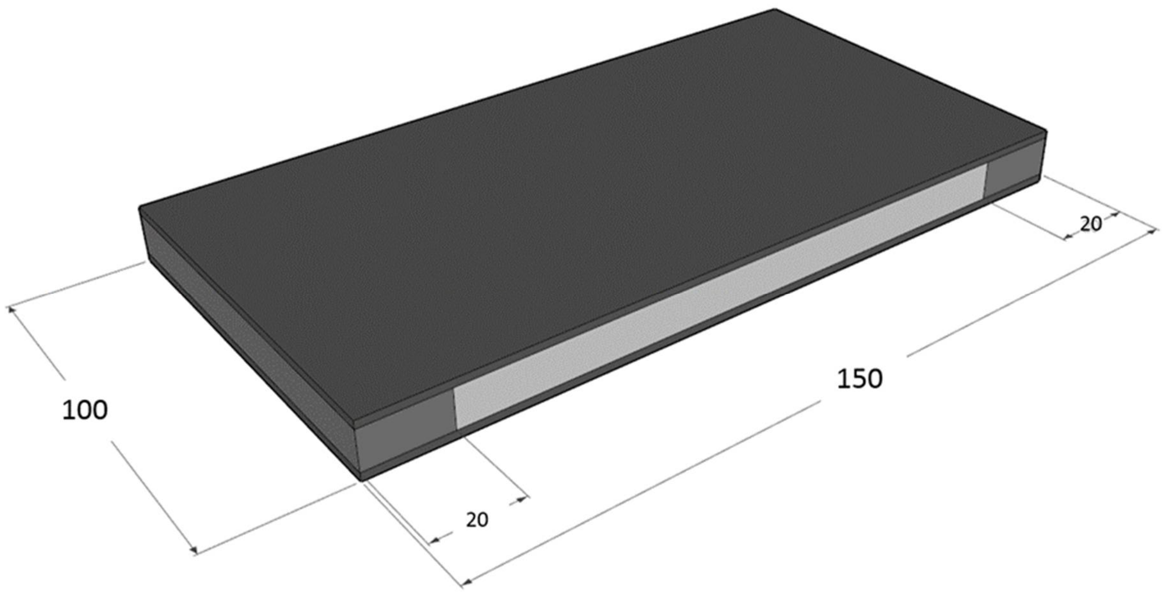

Figure 19: Test specimen geometry per ASTM D 7136/D 7136M – 07. Thickness 10 mm.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

TEST SPECIMENS CONSTRUCTION

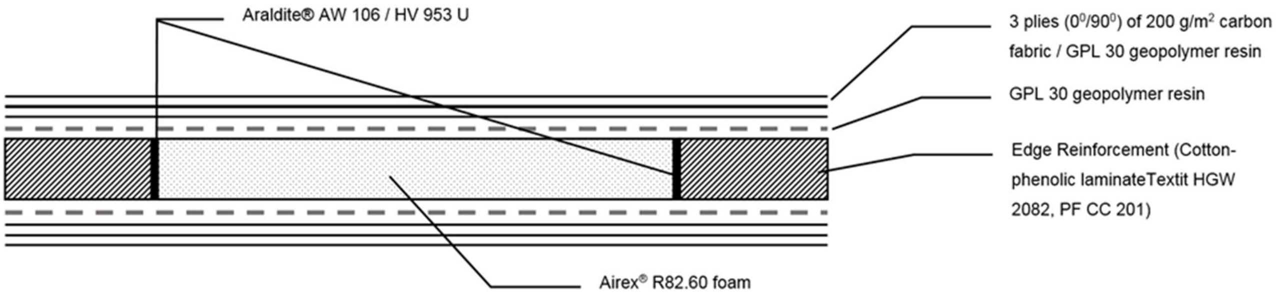

Figure 20: Lay-up arrangement of test specimens Type 1 (carbon/geopolymer)

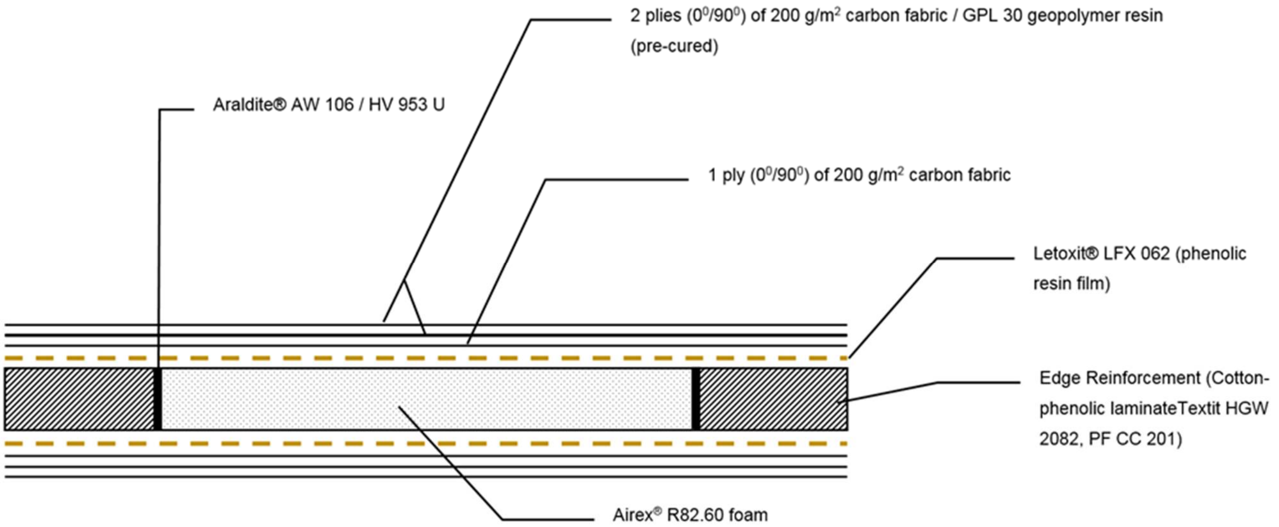

Figure 21: Lay-up arrangement of test specimens Type 2 (carbon/geopolymer/phenol hybrid)

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

Figure 22: Applied impact device

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

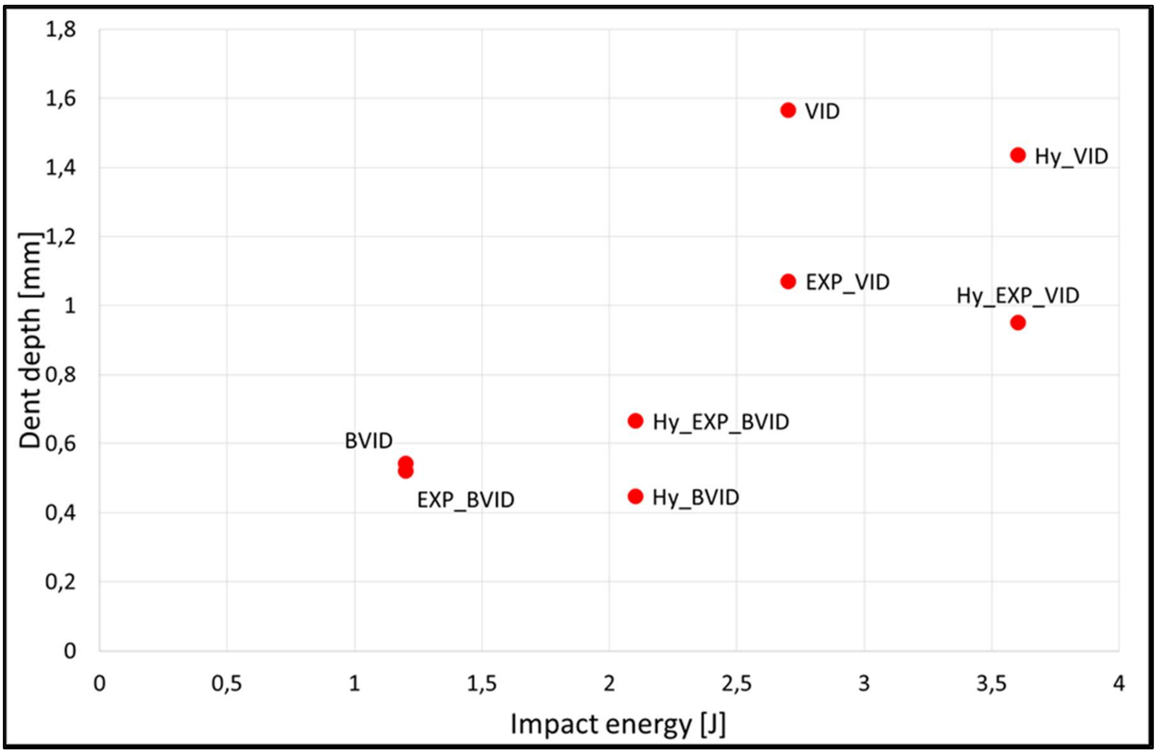

Figure 23: Functionality of impacts depth on impact energy for Type 1 and Type 2 („Hy“) skins

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

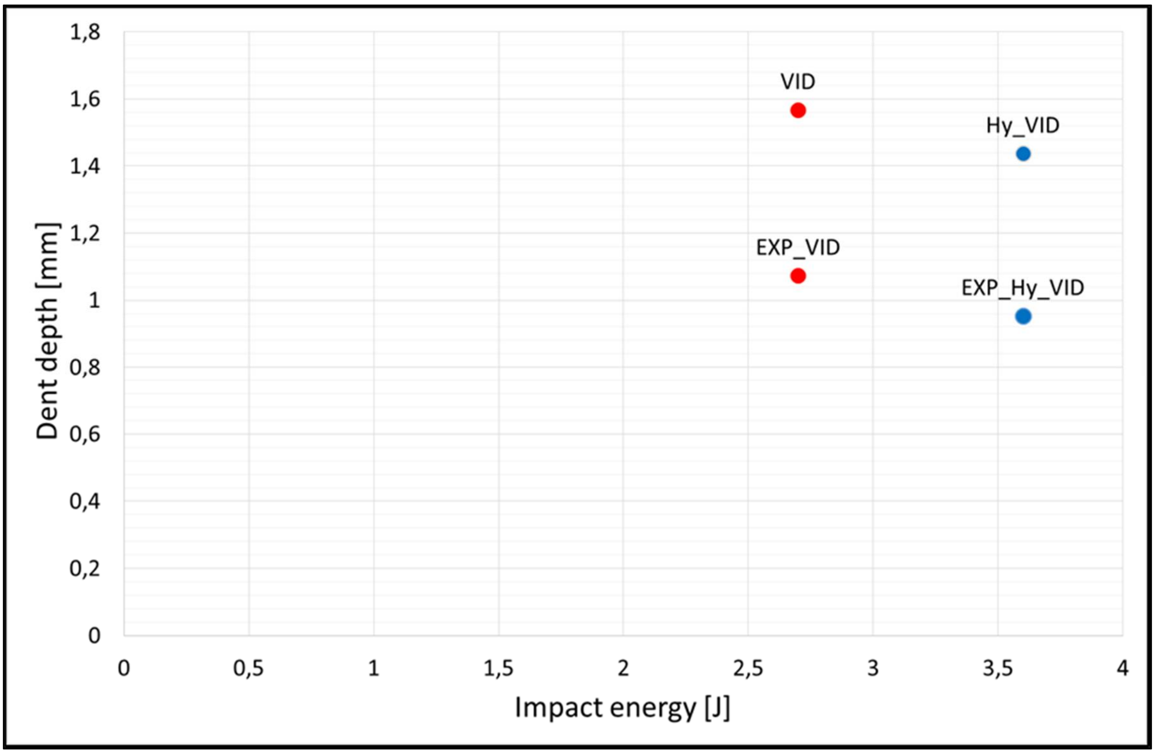

Figure 24: VID hot/wet exposed („EXP“) specimens showed approx. 30% better impact resistance compared to non-exposed ones

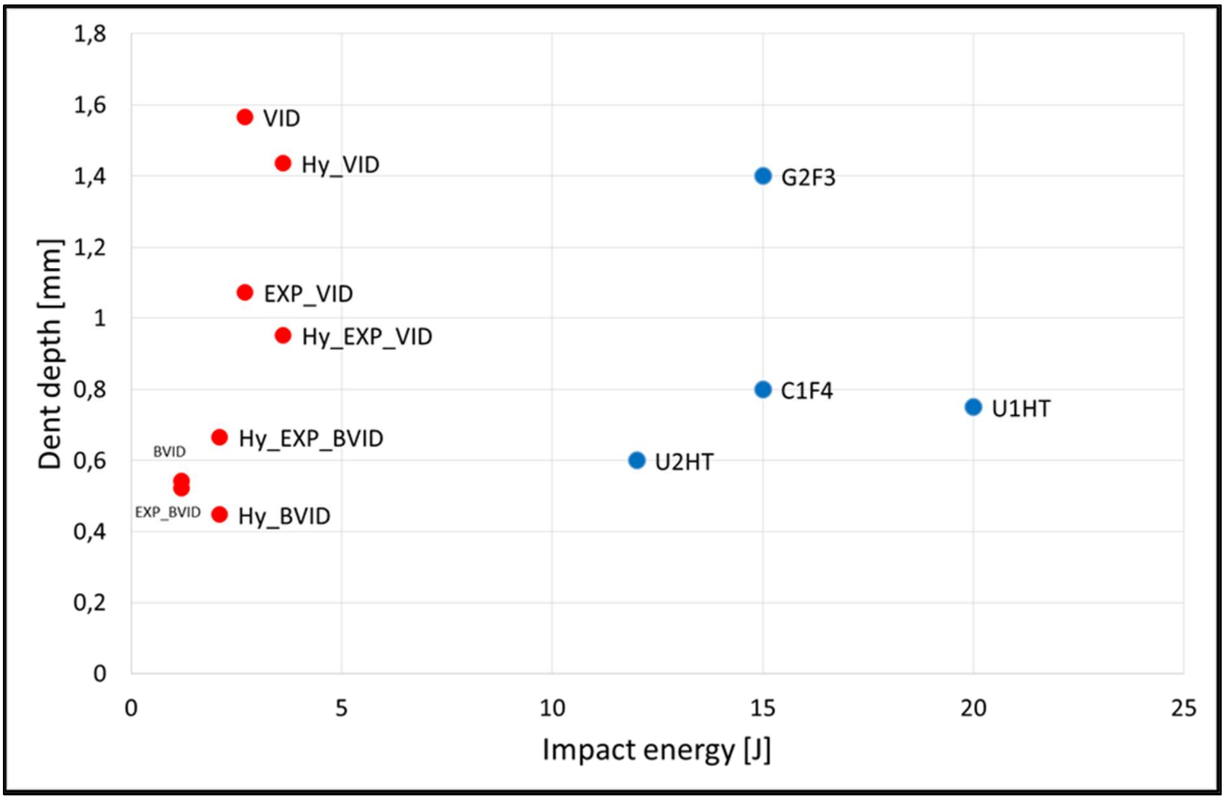

Figure 25: Comparison of impact resistance of Type 1 and Type 2 (red dots) specimens and referential specimens made of carbon/epoxy (blue dots)

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

3.1.4. Drum Peel Tests of Carbon Geopolymer Sandwich Panels

Peel strengths of sandwich panels constructed of carbon fiber reinforced geopolymer skins and honeycomb resp. foam cores were evaluated.

As referential group of specimens, GURIT PHG 600-68-37 (style 7781) glass/phenolic prepreg based, co-cured sandwiches were employed.

The tests were carried out in accordance with ASTM D1781 - 98 standard (Standard Test Method for Climbing Drum Peel for Adhesives), in normal conditions, with no previous environmental exposition.

MATERIAL COMBINATIONS TESTED

Skins:

a) 3 plies of 200 g/m2 carbon fabric / GPL 30 geopolymer resin

b) 3 plies of GURIT PHG 600-68-37 T2 (style 7781) glass/phenolic prepreg („REF“ specimens)

Adhesives:

a) Promat® K84 (anorganic adhesive)

b) Resbond® 989 (anorganic adhesive)

c) Letoxit® LFX 062 (phenolic film adhesive)

d) GURIT PH600-44-50 T2 (style 120 glass/phenolic prepreg)

e) GPL30 geopolymer resin

Cores:

a) EURO-COMPOSITES® ECA 4,8 – 48 (aramid honeycomb)

b) 3AComposites Airex® R82.60 (thermoplastic polymer foam)

Detailed information and unabridged test results are stated in the VZLU report No.: R-6724/2017.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

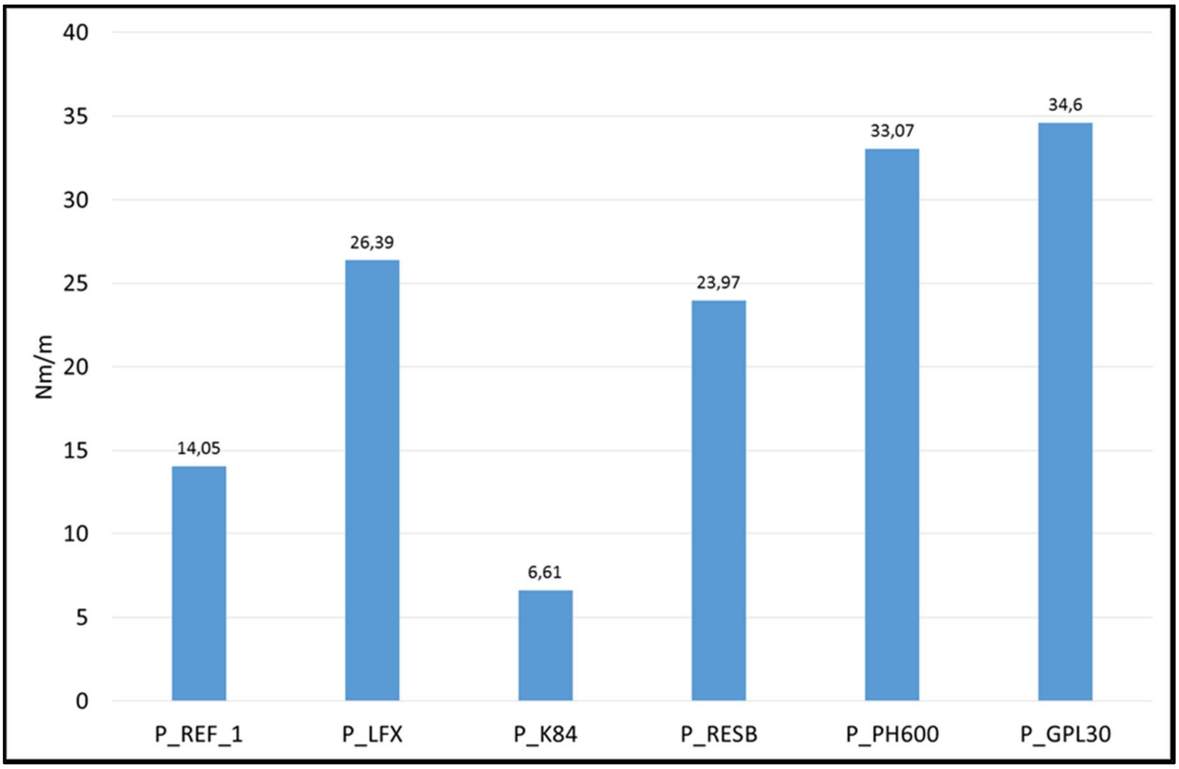

Figure 26: Average peel strengths of foam core specimens

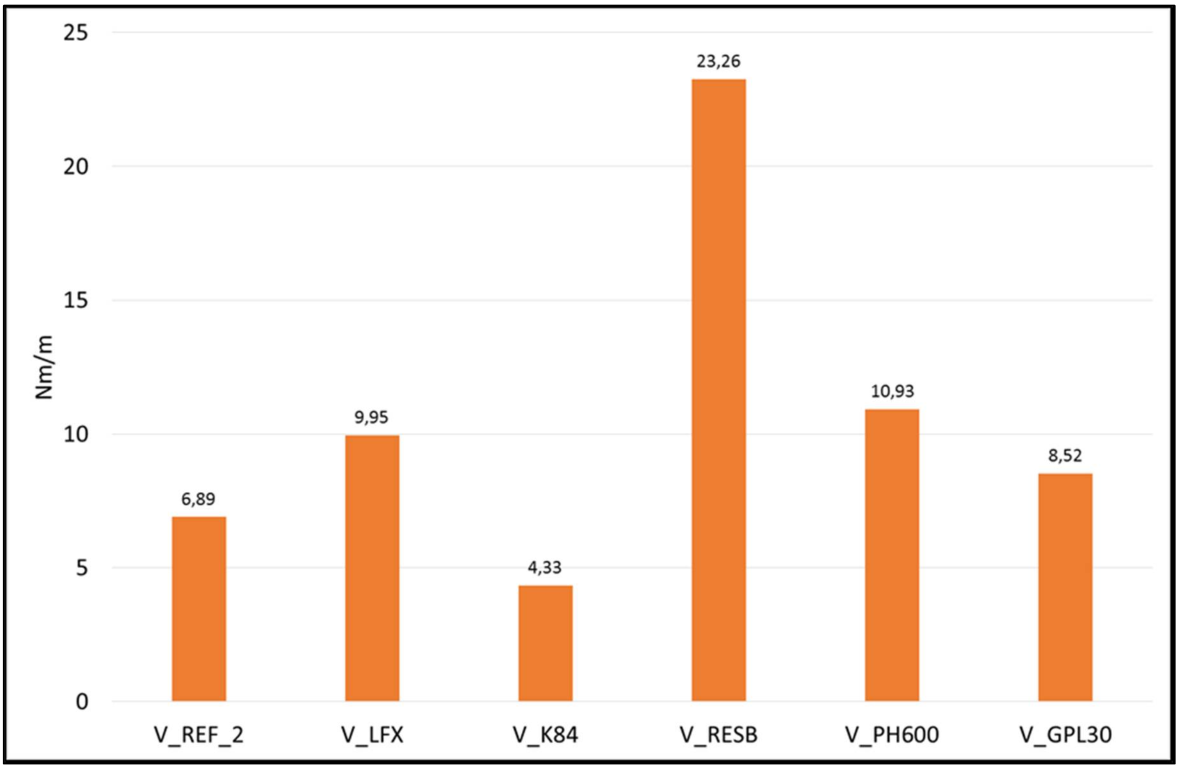

Figure 27: Average peel strengths of honeycomb core specimens

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

3.1.5. Compression Test of the Geopolymer Foam

Compression strength tests of the geopolymer hard foam were carried out.

The tests were carried out in accordance with ASTM D1621 standard.

Detailed information and unabridged test results are stated in the VZLU report No.: R-6810/2017.

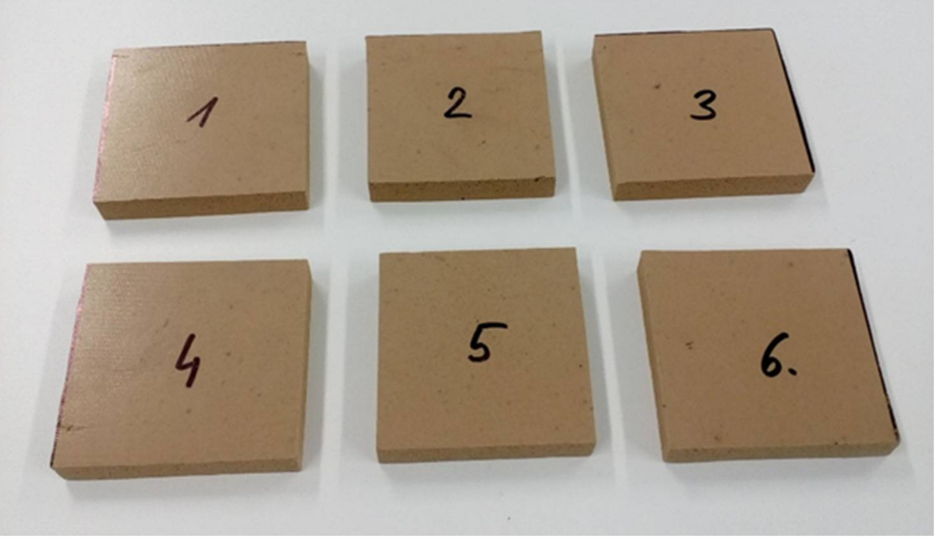

Figure 28: Test specimens (50 x 50 x 10 mm)

Figure 29: Detail of the geopolymer foam structure

Table 3: Specific weight and compression strength mean values of geopolymer foam specimens. Compared to referential Airex® thermoplastic foam.

	specific weight [kg/m3]	compression strength [MPa]	test standard
geopolymer foam	421	2,1	ASTM D1621
Airex R82.110	110	1,4	ISO 844

CONCLUSIONS

Fire Effluents and Smoke Optical Density of Carbon Fiber Geopolymer Composite:

From the point of view of criteria under review, carbon fiber reinforced geopolymer gives better results in comparison with referential glass/phenol in all evaluated parameters.

Flame Penetration Test per CS 25 Appendix F Part III:

No fire penetration was indicated both on carbon/geopolymer and referential glass/phenolic panels, regardless of the sandwich core material.

Temperature measured 102 mm (4") above the upper surface was exceeded in case of glass/phenolic panel.

Referential glass/phenolic panels have ruptured during the test resulting in smoke effluents escape from burned core material. No mechanical damage or ruptures and strong "pillow effect" were indicated on carbon/geopolymer panels.

Impact Tests of Carbon/Geopolymer and Carbon/Geopolymer/Phenol Sandwich Panels:

Visible Impact Damage (VID) and Barely Visible Impact Damage (BVID) were evaluated.

In the VID mode both TYPE 1 (geopolymer only skins) and TYPE 2 (hybrid skins) specimens surprisingly show better impact resistance after hot - wet exposition than not-exposed specimens. Possibly it is an attribute of additional post-curing of geopolymer matrix during the exposition in the climatic chamber.

In the BVID mode TYPE 1 specimens showed practically no sensitivity to hot - wet exposition. TYPE 2 specimens exhibit drop of impact resistance as expected.

Generally, TYPE 2 (hybrid skins) showed better resistance against the impact as presumed.

Comparison of of TYPE 1 and TYPE 2 specimens to referential specimens made of carbon/epoxy showed worse impact resistance of both geopolymers and hybrids.

Drum Peel Tests of Carbon Geopolymer Sandwich Panels:

In the group of foam core specimens the best results showed GPL 30 laminating resin bonded specimens, closely followed by PH 600 prepreg bonded samples.

In the group of honeycomb core specimens the best results showed Resbond® 989 bonded specimens, followed by PH 600 prepreg bonded samples.

Generally, foam core specimens provided better test results.

Compression Test of the Geopolymer Foam:

Development of the lightweight anorganic (geopolymer) foam is currently in the progress. Compression strength is comparable with referential Airex thermoplastic polymer foam. Test specimens of specific weight of cca 200 kg/m^3 are being tested.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

3.2. Eco-fibres with Geopolymer matrix

For the second test batch, flax fibre plain fabric and a nonwoven from recycled carbon fibres have been combined with Geopolymer (GPL) matrix (see chapter 2.2 for details of the used fibre materials). This combination aims to reduce the ecological impact of cargo compartment lining panels compared to State-of-the-Art materials made of glass fibres and phenol formaldehyde resin (GF-PF). Therefore tests to assess the basic fire properties (FST+HR) and mechanical properties (flexural strength and stiffness) have been conducted by DLR.

3.2.1. Composite manufacturing

The panels have been manufactured by project partner VZLU in Prague with flax fabric and rCF nonwoven provided by DLR. Hand laminating of each layer and a press process are shown exemplary in Figure 30: Manufacturing of composites with Geopolymer matrix (VZLU)

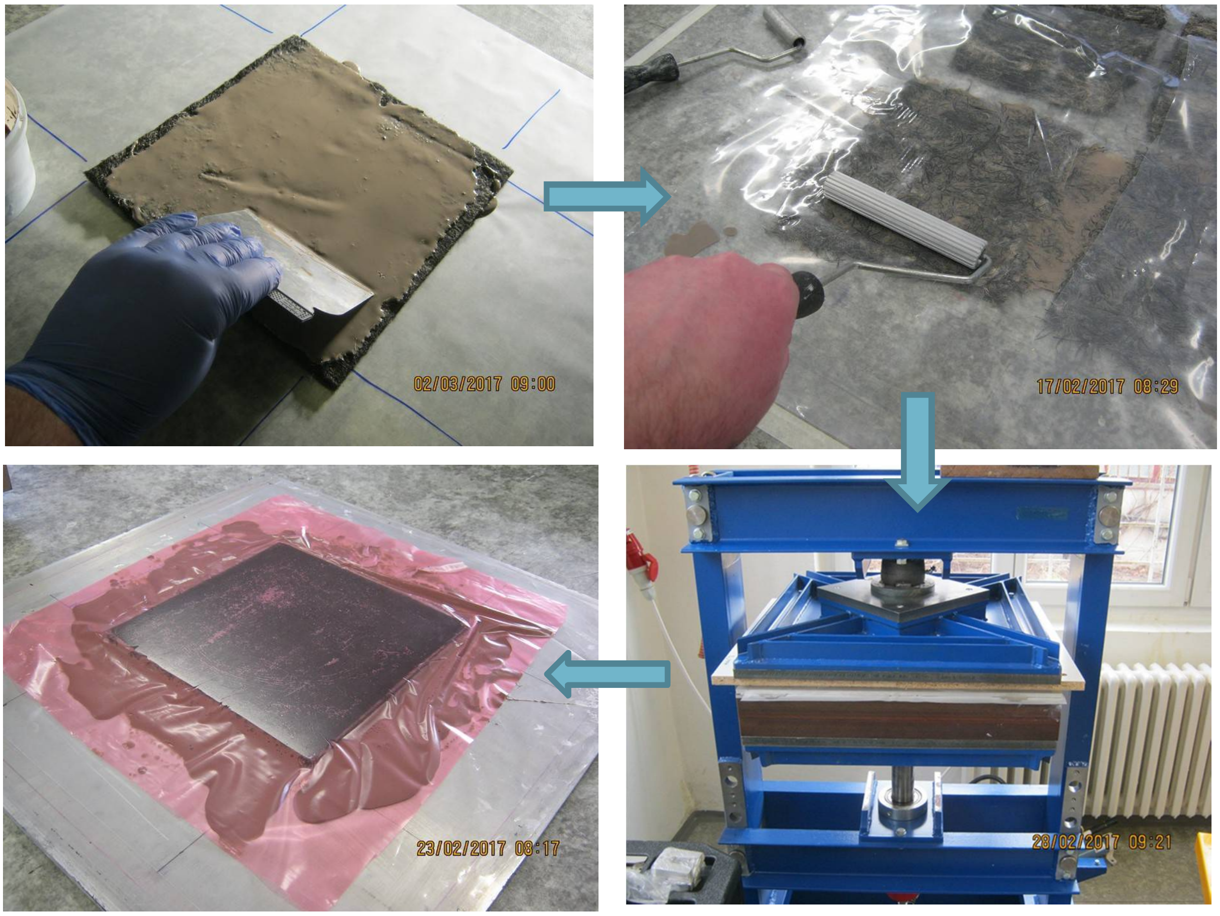

Figure 30: Manufacturing of composites with Geopolymer matrix (VZLU)

The parameters for all composites are shown in Table 4. For the fire tests, thin composites with a maximum of three layers have been manufactured in order to stay close to the reference panels made of three layers of glass fibre phenolic resin prepreg used as reference for the fire tests (plate number "NC123" in Table 4). As the geopolymer matrix has a comparatively high density of 2,54 g/cm³, it is of high

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

importance to use fibres with a reduced density compared to glass fibres (2,5 g/m³). Natural fibres (approximately 1,5 g/cm³) and carbon fibres (1,8 g/cm³) are therefore beneficial as reinforcements of geopolymers when compared to classic glass fibre composites. Furthermore, the typically high pore content of geopolymer composites reduces their density to lower values than calculated. Theoretically calculated values for Fibre Volume Content (FVC) and density based on aimed thickness and number of reinforcement layer are shown in Table 4. The actual measured values are shown, too. Based on the measured thickness and density of the composites, the actual FVC, density and pore content have been calculated.

Generally, three variants have been chosen: 100 % flax reinforcement, 100 % rCF reinforcement and hybrid variant with rCF outer layers and flax inner layers. The reason to use rCF as outer layer is twofold: First, for bending stress, the outer layers of rCF could profit from the higher mechanical properties compared to flax. Secondly, the outer layers of rCF could act as a protective layer regarding moisture uptake and fire properties for the more sensitive natural fibres.

Additional to the thin fire testing specimens, composites with a thickness of 4mm have been prepared to measure the basic mechanical properties with flexural tests. The same variants have been produced at DLR with a matrix from classic epoxy resin to compare the mechanical properties with geopolymer variants. The test results of the epoxy variants and also the hybrid geopolymer composite are not available before January 2018 and will be included in the annex of the final report in (Deliverable D7.13, March 2018).

Manual impregnation with the liquid geopolymer matrix could lead to voids in the final composite, this was particularly observed for the combination with rCF nonwoven (Figure 31). The calculated pore content of the rCF nonwoven reinforced geopolymer is up to 33% and needs to be better controlled in future manufacturing trials in order to get a better picture of the performance potential. Furthermore, a one-directional expansion or flowing of material has been observed for the 4mm composite because of the applied pressure in the hydraulic press, resulting in a reduced FVC of the test specimen (Figure 32).

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

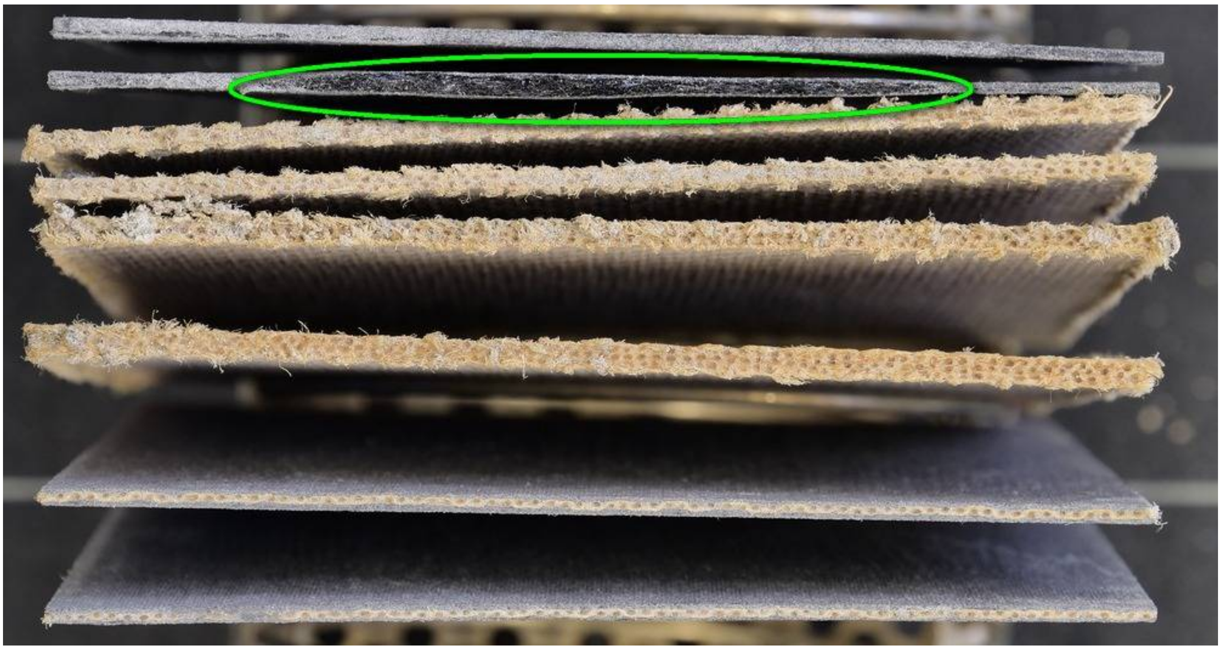

Figure 31: Dry area visible in the centre of a smoke density test specimen reinforced with 2 layers of rCF nonwoven and Geopolymer matrix (specimen side length 73mm, thickness approximately 1mm)

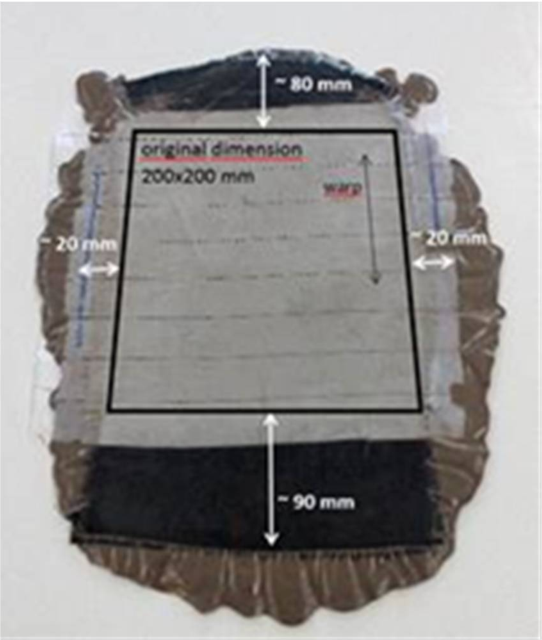

Figure 32: Expanded surface area (mainly in nonwoven machine direction (MD)) observed during the production of rCF nonwoven reinforced Geopolymer (20 layers) in a hydraulic press

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

Table 4: Parameters and measured values of second test batch specimens for the combination of eco-reinforcements (flax, rCF) with Geopolymer matrix (reference: Glass fibre (GF) with Phenolic Resin (PF)

DLR internal plate No.		Layup	Matrix	*FVC target* [%]	*Density target* [g/cm³]	*Thickness target* [mm]	Measured and calculated values from composite: **Thickness** *actual* (mean) [mm]	weight [g]	**Theor. Density** *actual* [g/cm³]	**Density** *actual* [g/cm³]	calc. **FVG** *actual* [%]	calc. **Pore content** [%]
NC117	FST+HR	2x rCF nonwoven (100g/m²)	GPL	20,0	*2,168*	*0,6*	0,99	357,1	2,20	1,45	12,2	33,5
NC118	FST+HR	2x Flax plain fabric (300g/m²)	GPL	54,8	*1,816*	*0,8*	1,09	415,0	1,93	1,53	40,4	17,6
NC119	FST+HR	3x Flax plain fabric (300g/m²)	GPL	59,8	*1,776*	*1,1*	1,47	543,0	1,90	1,49	44,9	17,9
NC120	FST+HR	***Hybrid*** 2x rCF nonwoven (100g/m²) (2 outer) + 1x Flax plain fabric (300g/m²) (inner)	GPL	37,7	*2,001*	*0,9*	1,08	471,2	2,04	1,75	31,4	12,9
NC123	FST+HR	3x PHG600 GF- PF Prepreg (296g/m²)	PF	49,7	*1,820*	*0,8*	0,79	96,4	1,83	1,71	50,5	10,9
NC124	FST+HR	2x rCF nonwoven (100g/m²)	GPL	20,0	*2,168*	*0,6*	0,53	52,9	2,16	1,59	22,5	25,2
NC121	Mech. Testing	20x rCF nonwoven (100g/m²)	GPL	30,0	*2,122*	*4,0*	3,66	487,6	2,11	1,90	32,8	9,1
NC122	Mech. Testing	8x Flax plain fabric (300g/m²)	GPL	43,9	*1,905*	*4,0*	3,95	400,1	1,90	1,62	44,4	12,6
NC125	Mech. Testing	***Hybrid*** 5x rCF nonwoven (100g/m²) (2 outer) + 5x Flax plain fabric (300g/m²) (inner)	GPL	42,4	*1,969*	*4,0*	4,72	559,1	2,01	1,75	36,0	11,8

3.2.2. Fire Properties of eco-fibres in combination with Geopolymer matrix

Fire tests at DLR site Trauen have been carried out according to aviation standards given in Table 5. Flammability, Smoke & Toxicity and Heat Release test specimen have been prepared at DLR site Braunschweig from the composites produced by VZLU. Flame Propagation tests have been omitted in the second batch of tests because of the comparable large sample size of 406 mm by 610 mm. Two samples for each test have been prepared, examples of the specimen made of rCF nonwoven reinforced GPL matrix are shown in Figure 33 respectively Figure 34 for the flax plain fabric reinforced variant. The irregular surface of the composites due to the hand-laminating process can be observed for both types of reinforcements.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

Table 5: Overview of fire test standards for cargo compartment

Abbr.	Standard: FAR/CS	Test Description	Dimensions [mm]
F	25.853(a)	Flammability vertical 12s	75 x 305 ("at least")
ST	28.853(d) + ABD0031 (Tox)	Smoke & Toxicity	73 x 73 (±2)
HR	25.853(d)	Heat Release (Rate)	150 x 150 (+0/-2)
FP (?)	25.855(c)	Fire Penetration Test (Oil Burner)	406 x 610 (± 3)

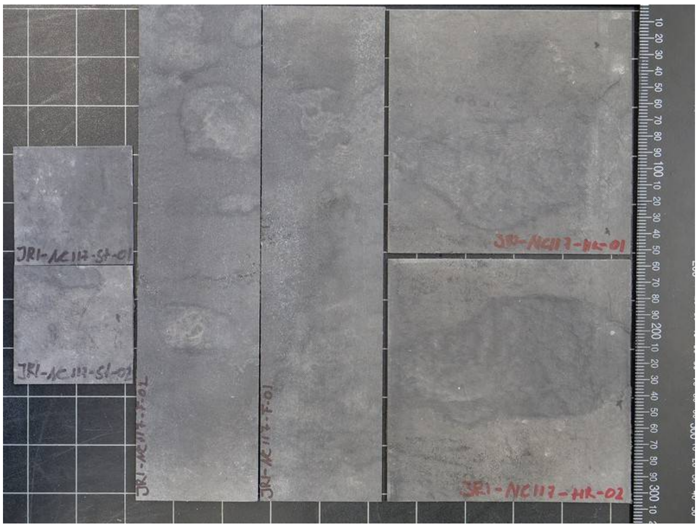

Figure 33: Samples for the F, ST and HR tests made of rCF nonwoven and Geopolymer matrix

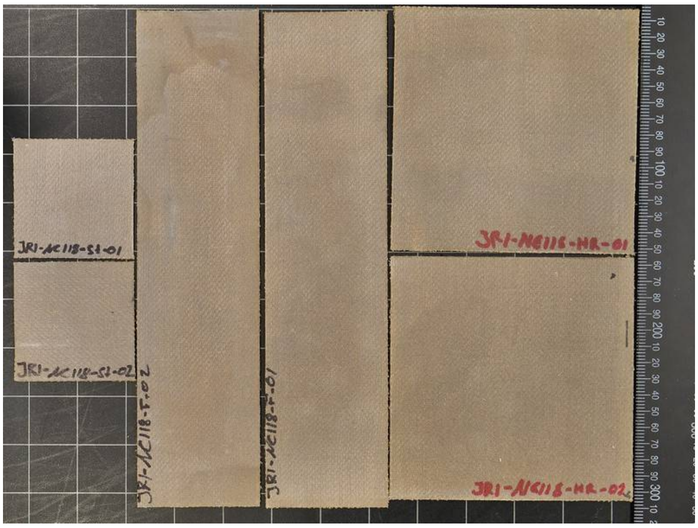

Figure 34: Samples for the F, ST and HR tests made of flax plain fabric and Geopolymer matrix

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

Test results for Flammability and Heat Release according to FAR 25.853 are summarized in Table 6: Test results of Flammability and Heat Release tests according to FAR 25.853 (rCF = rCF nonwoven 100g/m², Flax = Flax plain fabric 300g/m², GPL = Geopolymer, PF = Phenolic Resin, L = Layer)Table 6. The test results should be clearly below the limit values shown as red numbers in the table as potential decorative layers will typically add calorific value to the composite and therefore lead to higher flammability and heat release. A colour coding has been added to the background of the test values. Green shows a positive potential with a lot of room to the limit. It gradually changes to yellow and red as soon as the limit is reached or exceeded.

Table 6: Test results of Flammability and Heat Release tests according to FAR 25.853 (rCF = rCF nonwoven 100g/m², Flax = Flax plain fabric 300g/m², GPL = Geopolymer, PF = Phenolic Resin, L = Layer)

DLR internal sample No	Material	Flammability Vertical 12s			Heat Release		
		Burn Length	After Flame Time	Drip Flame Time	HRRmax		HR
		[mm]	[s]	[s]	[kW/m²]	at [s]	[kW*min/m²]
	Limit ->	*203*	*15*	*5*	*65*	*-*	*65*
NC117-1	2L rCF + GPL	17	0	0	8	277	3
NC117-2	2L rCF + GPL	24	0	0	11	286	6
NC124-1	2L rCF + GPL	1	0	0	7	260	5
NC124-2	2L rCF + GPL	1	0	0	8	281	6
NC118-1	2L Flax + GPL	19	0	0	195	35	78
NC118-2	2L Flax + GPL	28	0	0	184	34	75
NC119-1	3L Flax + GPL	9	0	0	244	48	110
NC119-2	3L Flax + GPL	8	0	0	264	48	115
NC120-1	2L rCF + 1L Flax + GPL	16	2	0	62	34	25
NC120-2	2L rCF + 1L Flax + GPL	20	0	0	82	35	27
NC123-1	3L GF + PF (Ref)	3	2	0	58	41	36
NC123-2	3L GF + PF (Ref)	1	2	0	60	41	36

For the flammability test, all tested samples show promising results and are roughly on the same level with the reference made of GF-PF prepreg (NC123). Nevertheless, the fluctuations (see error bars in Figure 36) of results are high and more samples need to be tested for a clearer picture. No dripping flame has been observed for all specimens. Only the hybrid variant and GF-PF reference show a short after flame time far below the limit of 15 seconds. Photographs taken during the flammability test show a comparable behavior of all samples (Figure 35).

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

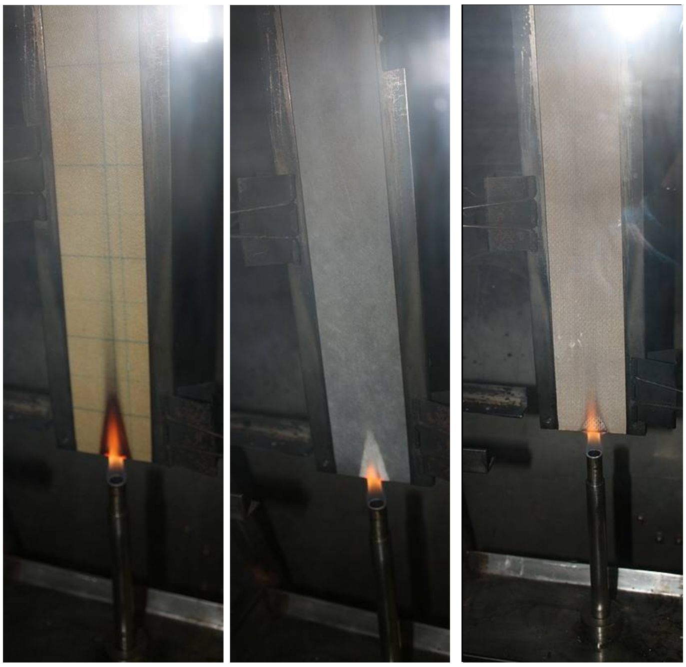

Figure 35: During the Flammability test (12s, vertical): GF+PF reference (left), rCF+GPL (middle) and flax+GPL (right).

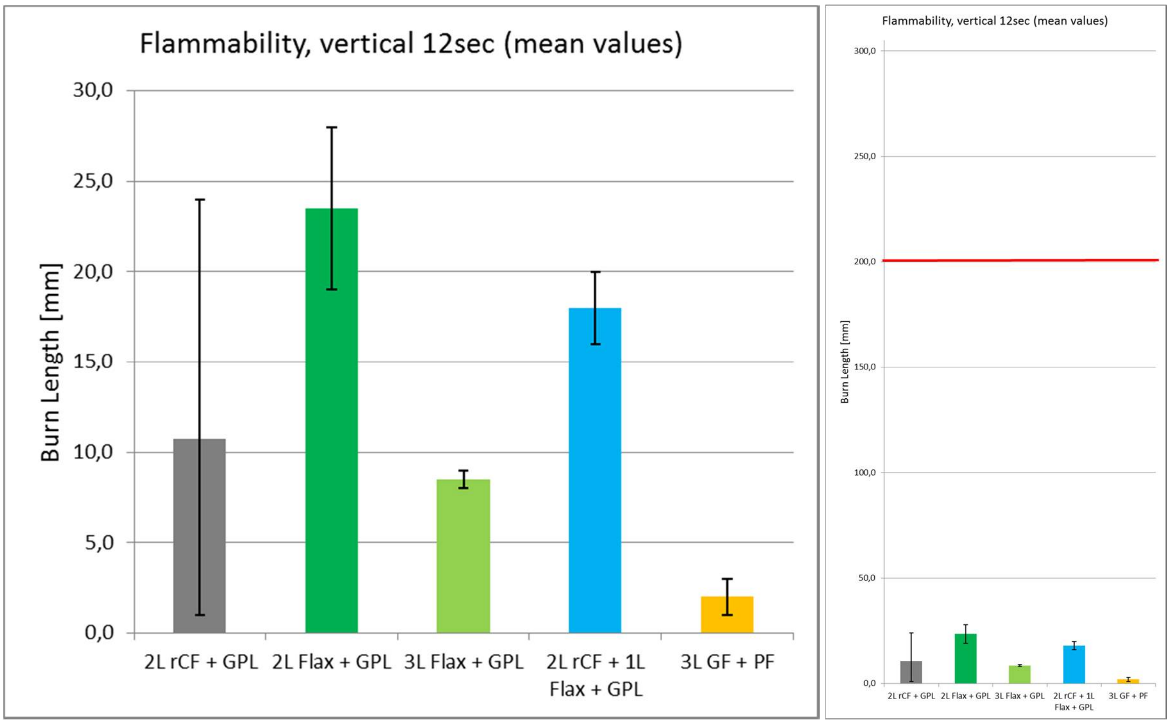

Figure 36: Flammability of the eco-fibres with GPL matrix and GF-PF reference according to FAR 25.853. The right hand figure shows the same results in relation to the specimen length. All results are far below the limit burn length of 203mm indicated by the red line.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

Heat Release (HR) and Heat Release Rate (HRR) show a different picture with a clearer distinction of the different reinforcement materials (Table 6, Figure 37). Here, the advantage of geopolymer matrix compared to phenolic resin can be observed despite the use of different reinforcement materials, as rCF and GF both show good fire properties. On the other hand, the cellulosic flax fibres show their negative impact on the heat release and especially the rate of heat release. The embedding in a fire resistant geopolymer matrix alone is not enough to protect the fire sensitive flax fibres. It has to be explored, if a better composite quality with reduced void content is able to improve the heat release results considerably. Otherwise the addition of a flame retardant is still needed to fulfil the demanding aviation requirements. Furthermore, the higher calorific value of three layers flax is observable in form of higher HR and HRR compared to the 2 layer variant.

A possible way to use eco-fibres could be a hybrid composite. The example of 2 rCF outer layers and one flax inner-layer shows comparable results to the reference GF-PF.

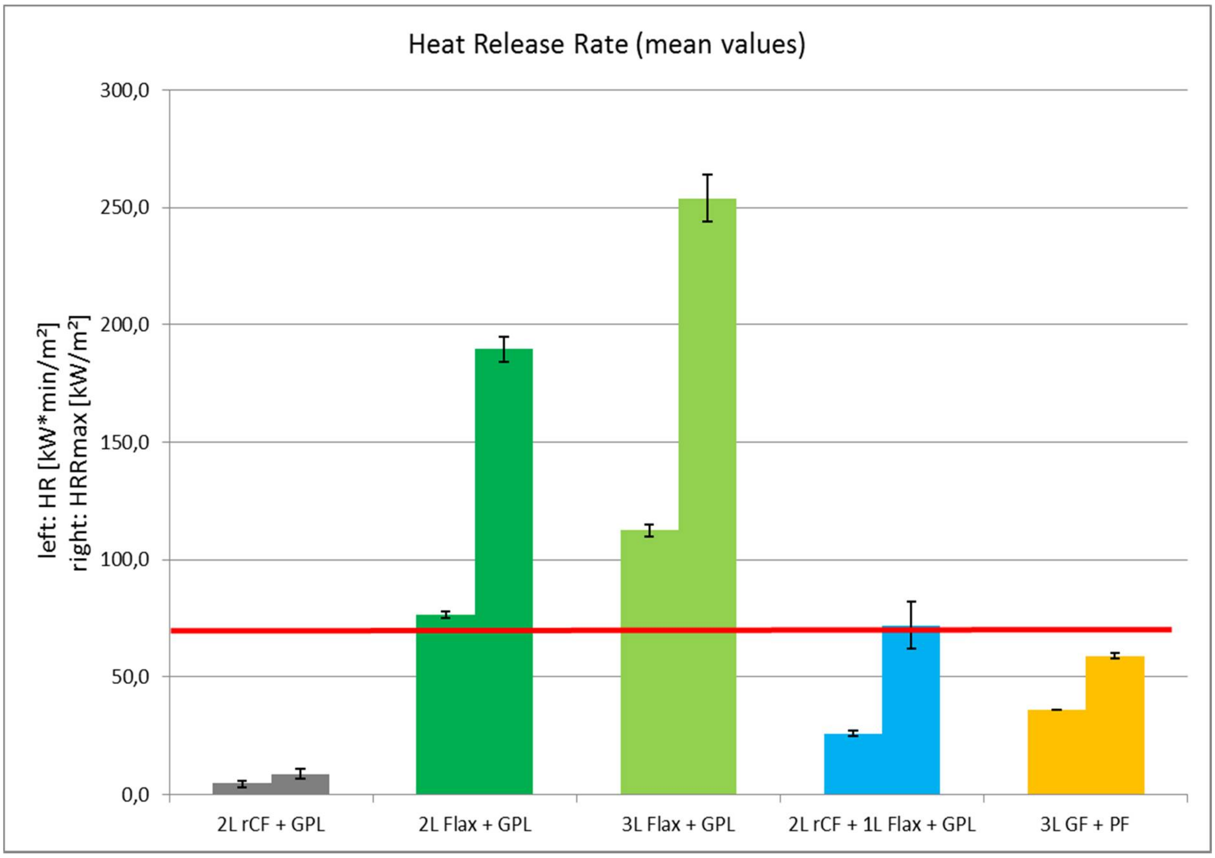

Figure 37: OSU results for Heat Release (left column for each material combination) and Heat Release Rate (right column) of the eco-fibres with GPL matrix and GF-PF reference according to FAR 25.853. The red line indicated the limit of 65 kW/m² respectively 65 kW*min/m².

Smoke Density (S) and Toxicity (T) tests are normally carried out together in one test. Samples reinforced with rCF show the lowest smoke density and toxicity, followed by the reference of GF-PF. The high potential of geopolymer resin to reduce fumes and toxicity in cabin environment is therefore shown.

Higher smoke density and toxicity values can be observed for the flax reinforced geopolymer. A higher amount of flax fibres (three layers compared to two) increases smoke density and toxicity for CO and SO2. However, the test results are still under the limit for cargo compartment liner application. Additionally conducted tests with pure flax fabric (without matrix) show comparable results for toxicity and a higher smoke density because of the missing protection by a matrix system.

Table 7: Smoke Density (S) and Toxicity (T) test results (rCF = recycled carbon fibre nonwoven 100g/m², Flax = Flax plain fabric 318g/m², GPL = Geopolymer, PF = Phenolic Resin)

Sample	Material	Smoke Density		Toxicity					
		Non-flaming	Flaming	<-- Mode					
		Ds	Ds	HCN	CO	NOx	SO2	HF	HCl
		[-]	[-]						
	Limit ->	*200*	*200*	*150*	*1000*	*100*	*100*	*100*	*150*
NC117-1	2L rCF + GPL	1		0	46	1	10	0	0,3
NC117-2	2L rCF + GPL		1	0	3	0	0	0	0
NC124-1	2L rCF + GPL								
NC124-2	2L rCF + GPL		1	0,1	60	0	23	0	0,1
NC118-1	2L Flax + GPL	12		0,5	385	6	59	0	0,5
NC118-2	2L Flax + GPL		15	0	352	3	30	0	0
NC119-1	3L Flax + GPL	19		0,5	513	7	80	0	0,2
NC119-2	3L Flax + GPL		38	0,1	576	6	54	0	0,1
NC120-1	2L rCF + 1L Flax + GPL	6		0,2	255	2	31	0	0,2
NC120-2	2L rCF + 1L Flax + GPL		4	0,1	174	2	15	0	0,1
NC123-1	3L GF + PF (Ref)								
NC123-2	3L GF + PF (Ref)		2	0,5	100	2	37	0	0
BL300-1	1L Flax Fabric (pure)	133		0,3	327	3	54	0	0
BL300-2	1L Flax Fabric (pure)		37	0,3	214	4	57	0	0,1

3.2.3. Mechanical properties of eco-fibres in combination with Geopolymer matrix

To assess the mechanical properties of the combination of eco-fibres with geopolymer matrix, a simple three point bending test according to DIN EN ISO 14125 has been carried out by DLR. Table 8 shows the properties of the composites produced for mechanical testing, containing eco-fibres reinforcing a geopolymer matrix. It is worth pointing out the high pore content between 9,1 % and 12,6 %. However, the pore content is lower compared to the composites produced for fire testing (see Table 4 for comparison). Specimens have been cut to a size of 80 mm to 10 mm. Three point bending tests have been carried out after storage at 23°C and 50% relative humidity for at least one week.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

Table 8: Composites from eco-fibres and geopolymer produced for mechanical testing

DLR internal plate No.		Layup	Matrix	*FVC target* [%]	*Density target* [g/cm³]	*Thickness target* [mm]	Measured and calculated values from composite: Thickness *actual* (mean) [mm]	weight [g]	Theor. Density *actual* [g/cm³]	Density *actual* [g/cm³]	calc. FVG *actual* [%]	calc. Pore content [%]
NC121	Mech. Testing	20x rCF nonwoven (100g/m²)	GPL	30,0	*2,122*	*4,0*	3,66	487,6	2,11	1,90	32,8	9,1
NC122	Mech. Testing	8x Flax plain fabric (300g/m²)	GPL	43,9	*1,905*	*4,0*	3,95	400,1	1,90	1,62	44,4	12,6
NC125	Mech. Testing	***Hybrid*** 5x rCF nonwoven (100g/m²) (2 outer) + 5x Flax plain fabric (300g/m²) (inner)	GPL	42,4	*1,969*	*4,0*	4,72	559,1	2,01	1,75	36,0	11,8

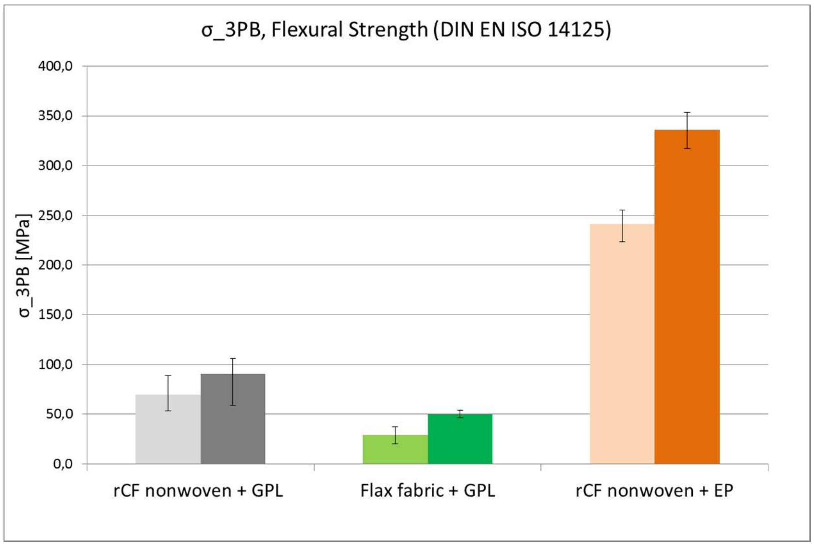

Figure 38: Flexural Strength

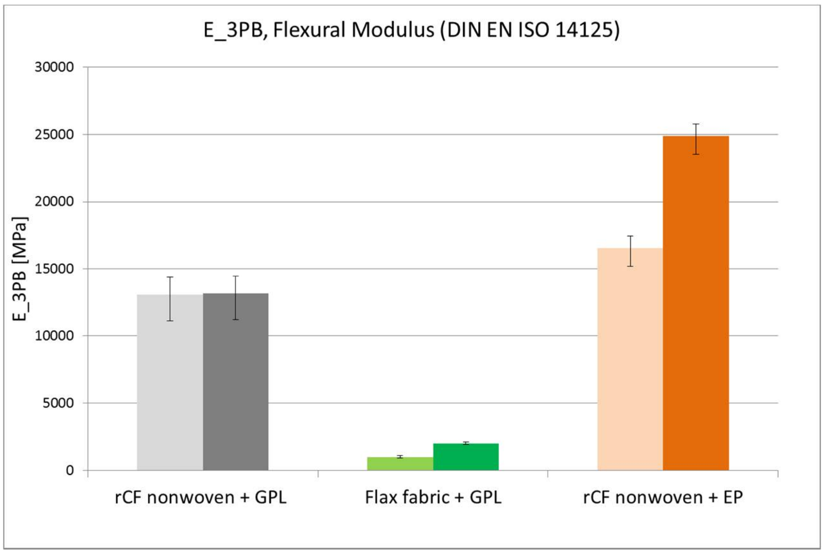

Figure 39: Flexural Modulus

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

The results of the flexural tests are shown in Figure 38 for flexural strength and Figure 39 for flexural modulus. So far, only 100 % flax fibre reinforced geopolymer and the 100 % rCF reinforced geopolymer have been tested. Results of the hybrid variant and reference samples with epoxy resin will be added to the annex of the final report D7.13. For comparison, further results of rCF nonwoven with epoxy resin are included to the figures.

Very low flexural strength and modulus can be observed for the flax fibre reinforced geopolymer. An evaluation is difficult without macroscopic and SEM images of the samples. Images of the tested samples do not show any visible fracture Figure 41. The flax + GPL samples show a considerable amount of plastic deformation with a strain at maximum force around 10 %. During testing, a fracture is visible (Figure 40). Generally, the test results are very weak with a mean flexural strength of 50 MPa, respectively 1989 MPa flexural modulus in 90° test direction. Test in 0° direction show even lower results. Possible measures to improve the mechanical properties of flax reinforced geopolymer will be discussed in the conclusions and recommendations in Chapter 4.

Figure 40: Considerable deflection of flax fabric + GPL sample during flexural testing

Figure 41: Samples after testing (flax fabric + GPL, left = 0°, right = 90° test direction)

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

The geopolymer matrix normally shows a brittle behaviour. This is visible for the recycled carbon fibre nonwoven reinforced variant (rCF + GPL) in Figure 42. Compared to the flax+GPL variant, the results are considerably higher, nevertheless they are still lower compared to epoxy matrix. The carbon fibres pulled out of the samples in Figure 42 indicate a weak fibre matrix adhesion as possible explanation.

Figure 42: Fracture of rCF+GPL sample after flexural test (side view)

Further test results will be included to the annex of D7.13 (March 2018).

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

3.3. Fibre Metal Laminates

All tests of FML that are shown in the following are conducted with specimens 4-3950-125 with a laminate of $[[0°/90°]\ St\ [0°/90°]\ St\ [0°/90°]]_S$. Each unidirectional cfrp ply and the steel layers have a thickness of 0.125mm cumulating to a laminate thickness of 2.0mm. The manufacturing process is described in D7.5. The results are compared to results of cfrp specimens based on a fully unidirectional layup.

3.3.1. Compression under fire test

3.3.1.1. Test setup

To test the mechanical properties of materials under conditions of a fire scenario, a new test facility was established. A hydraulic press was enhanced by a specimen device that contains a fire load withstanding clamping mechanism. The clamping is conducted by a potting of concrete material that is located inside a steel mould. The concrete potting material clamps the specimen against out-of-plane deformation. In-plane compression loads are applied through the face of the mould. To ensure parallel load introduction, the moulds are connected by installation struts. Additionally, the struts are temporarily enhanced by adjustment supports allowing precise specimen adjustment inside the first mould. The mould with the adjusted specimen is filled with the concrete material. After cure of the concrete material, the adjustment supports are removed and the second mould is applied to the installation struts and the specimen. Again, concrete material is filled into the mould and cured. The installation struts are dismantled at the end of the mounting procedure. The tested specimens have a dimension of 200mm length and 120mm width. A length of 40mm at each side is located inside the potting and thus the specimen field exposed to fire will have quadratic dimensions of 120mm side length. An additional aperture is available to reduce the area that is exposed to the flames. The specimens are curved to avoid buckling. The Cufex test stand is shown in Figure 43, a test specimen device is shown in Figure 44. Three pairs of potting moulds are manufactured. The mounting inside the press is a relatively fast process. This allows to test up to three specimens at a single test day. The test procedure and results are described in section 3.3.2.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

Figure 43: CuFex test facility

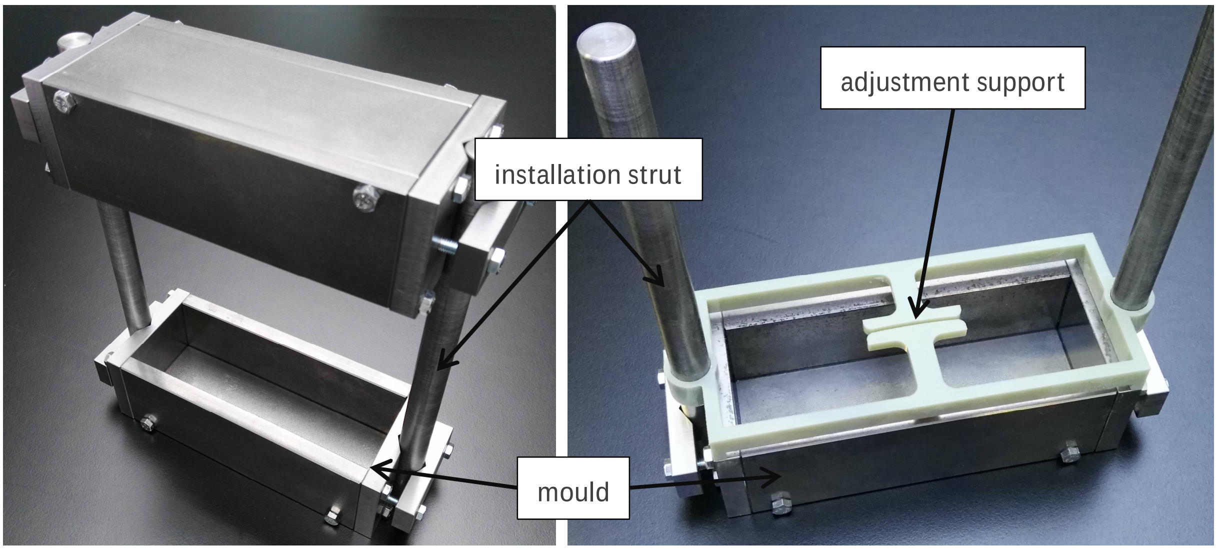

Figure 44: Specimen device; Installation struts and adjustment support are removed after moulding of the specimen

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

3.3.1.2. Test accomplishment

The clamped specimen is loaded by a compression force of 12kN. For the cross section of used specimens (120mm width, 2mm thickness) this equals a stress magnitude of 50MPa. The compressive load is a preload that is kept constant within until structural collapse is reached. A burner calibration that contains heat flux density and temperature measurement is conducted to ensure comparability of applied fire loading. After that, the mechanically preloaded structure is exposed to the calibrated burner fire. The fire load is kept until the specimen fails mechanically. An overview to the test procedure is shown in XXX. The tested specimens and test conditions are listed in **Table 9**:

Table 9: Overview to conducted CuFex-Tests

No	specimen	test description
1	FML-00	- Initial test to ensure test stand functionality - specimen with manufacturing defects used - complete free specimen exposed to fire
2	FML-01	- local fire exposure (40x40mm aperture)
3	FML-02	- local fire exposure (40x40mm aperture)
4	FML-03	- local fire exposure (40x40mm aperture)
5	FML-04	- local fire exposure (40x40mm aperture)
6	GP-01	- local fire exposure (40x40mm aperture)
7	FML-05	- local fire exposure (40x40mm aperture)
8	CFRP-01	
9	GPL-01	

In all tests the axial force was measured as well as the axial shortening. The temperature was measured at the rear side center of the structure. To this a spring mechanism was used to stich a thermocouple with a small force onto the specimen surface. This method is used to avoid effects due to adhesive connection.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

The first specimen FML-00 had several manufacturing defects and was used to validate the working principle of the CuFEx test stand. The test was performed without an aperture in front of the specimen. Hence the entire width of the specimen was heated. This resulted in a short life after the flame was applied to the specimen. Furthermore, the desired pillow effect was not visible. Due to this, the first test was excluded from further investigation.

The second and third specimen (FML-01 and 02) were tested with an aperture to reduce the exposed area to 40x40mm². This resulted in a longer specimen life and the development of a "pillow" on the back side of the specimen. The photo in Figure 45 shows this pillow and also leaking gases from the specimen.

The graphs in Figure 45 show the force that was applied and held and temperature on the back of the specimen. Around 20s after the start of the test, the flame was applied to the specimen. This resulted in a rising temperature and an expansion of the specimen, which lead to a slightly rising force. Shortly after that, the pillow started to develop on the back and inside the specimen. These pillows seem to have a strong internal pressure, as the leakage of the combustion gases lead to a noticeable jerk in the force measurement.

The specimens withstood the mechanical load even after the pillows leaked and collapsed after more than 90s of flame exposure.

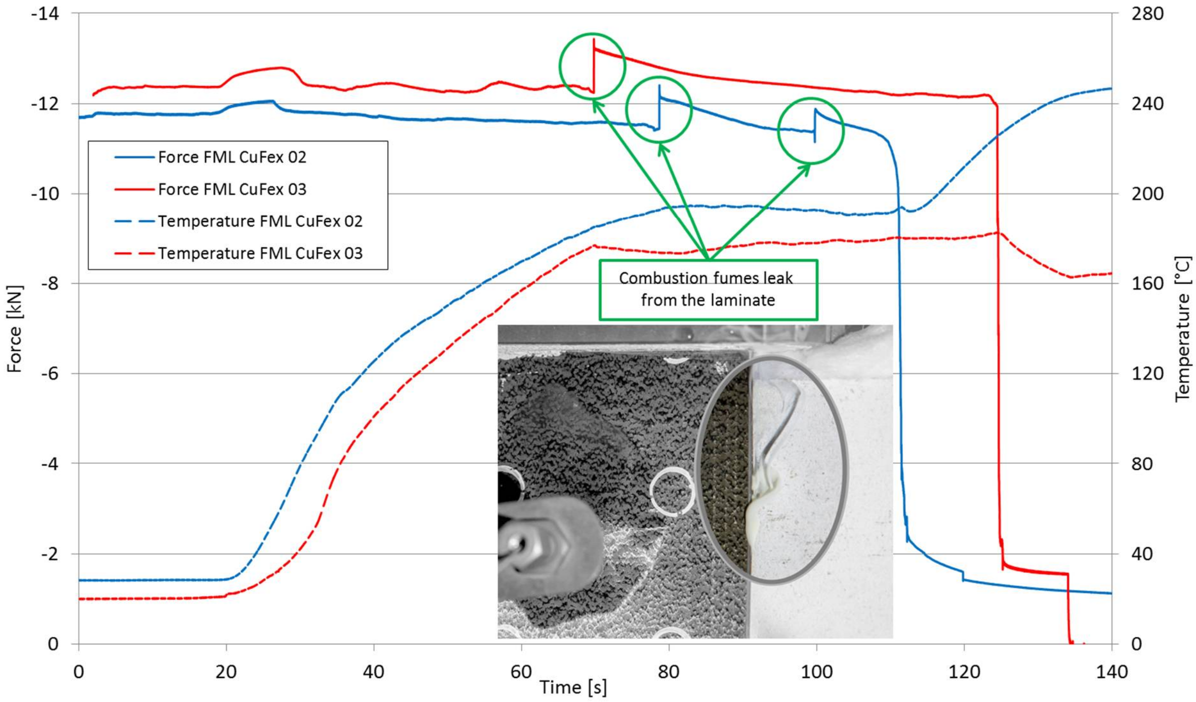

Figure 45: Force and temperature curves of two exemplary CuFEx-specimens

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

3.3.2. Material modelling

3.3.2.1. DMA

To gather the temperature dependent material properties DMA measurements are conducted. The DMA measurement was done in bending mode. Since the FML layup contains 0° and 90° oriented unidirectional cfrp layers specimens of all three types are tested. The results are shown in Figure 46

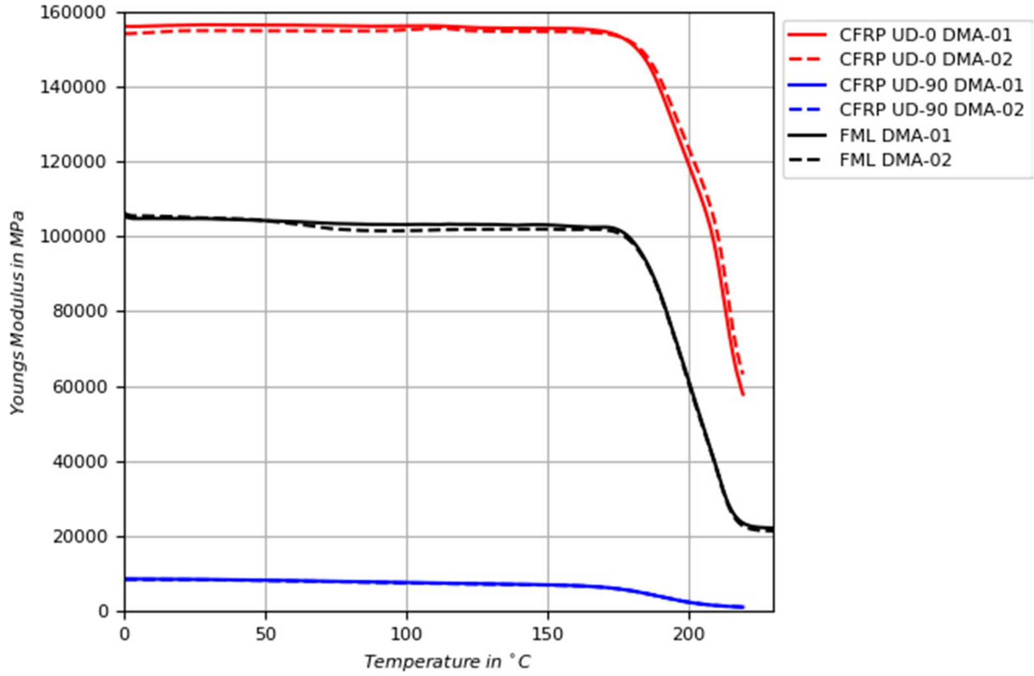

Figure 46: DMA results of FML and its cfrp components

3.3.2.2. Shear properties

Shear tests are conducted according to ASTM D 5379. Both, unidirectional cfrp and FML were tested at RT, 100°C, 150°C and 180°C. The test results are shown in Table 10 and Figure 47.

Table 10: Results of shear tests according to ASTM D 5379

	CFRP UD 0°-oriented		FML	
	Shear Modulus	Shear strength	Shear Modulus	Shear strength
RT	6609.5±358.2MPa	77.9±8.0MPa	22126.5±960.9MPa	250.8±10.1MPa
100°C	4639.7±66.3MPa	56.4±3.5MPa	14195.2±1110.6MPa	207.2±12.8MPa
150°C	3358.1±233.4MPa	31.6±1.6MPa	13567.1±2766.1MPa	171.1±9.4MPa
180°C	1353.1±400.4MPa	13.7±0.5MPa	10066.8±334.5MPa	100.6±6.3MPa

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

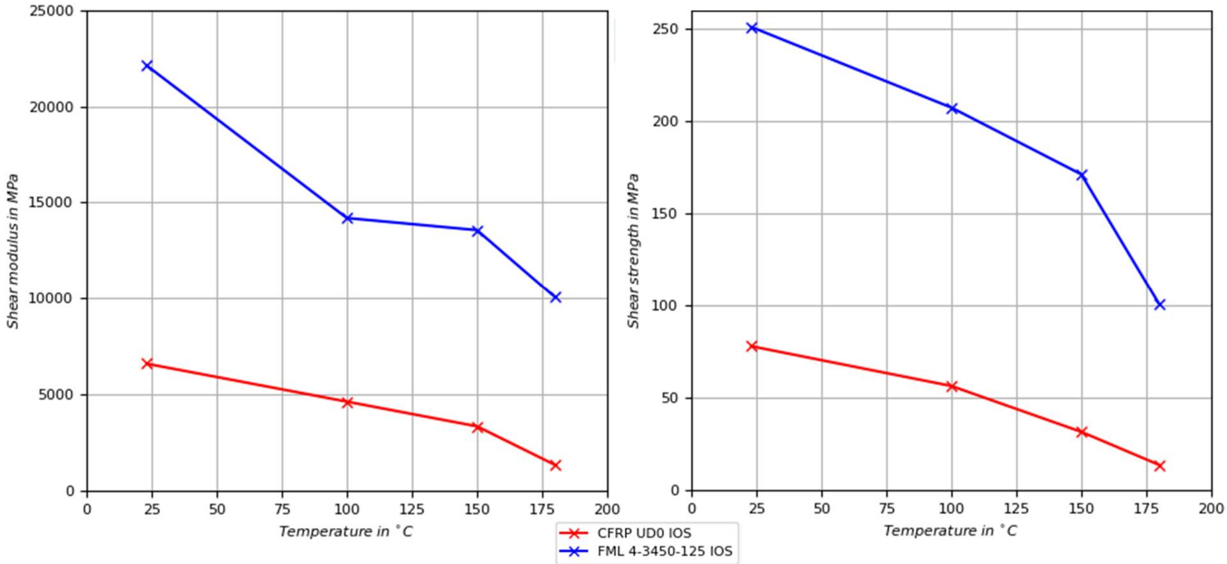

Figure 47: Temperature dependent shear properties of FML and cfrp from test results according to ASTM D 5379

3.3.2.3. Compression properties

Compression tests are conducted according to DIN EN 2850. The method differs between specimens to measure the compression modulus (no tabs but strain gauge) and specimens to measure the compression strength (with tabs bot no strain gauge).

Table 11: Results of compression tests according to DIN EN 2850

temperature	FML Modulus	FML Strength	CFRP UD Modulus	CFRP UD Strength
RT	102.89±0.94GPa	521.3±35.5MPa		861.2±59.4MPa
100°C		482.6±34.1MPa		788.8±40.6MPa
150°C		416.6±26.6MPa		602.2±14.7MPa
180°C		239.9±19.3MPa		269.3±8.2MPa

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

4 CONCLUSIONS AND RECOMMENDATIONS

4.1. Geopolymers

In all evaluated parameters of fire effluents & smoke optical density and flame penetration resistivity the carbon/geopolymer composite featured excellent test results and highly exceeded referential glass /phenolic material.

In case of the impact properties, both evaluated carbon/geopolymer and carbon/geopolymer/phenol hybrid composites appeared as more brittle compared to referential carbon/epoxy specimens. The energy amount for creation of comparable impact damage was approximately three times lower in case of carbon/geopolymers compared to the epoxy based test panels.

Concerning the peel strength of sandwich specimens with carbon fiber / geopolymer skins, geopolymer laminating resin GPL30 resulted as the optimal adhesive for foam core structures. In case of honeycomb core panels, Resbond® 989 anorganic adhesive provided the best results, but relatively high specific weight of the Resbond® adhesive have to be considered.

Development of the lightweight anorganic (geopolymer) foam is currently in the progress. Compression strength 2,1 MPa of 420 kg/m3 foam has been achieved. The current effort is focused to reduction of the foam specific weight.

More research effort should be focused on improvement of toughness of geopolymer based thin-wall composites. Combination of brittle carbon fiber with brittle geopolymer matrix doesn't seem to be optimal. Incorporating ductile fibers featuring good fire resistivity (para-aramids) could be the solution. Application of e.g. carbon/aramid hybrid reinforcement instead of carbon only fabric may improve impact resistivity significantly. This way of solving, due to lightweight aramid, may reduce the composite specific weight, too.

Utilization of resilient hybrid matrix systems (geopolymer/phenol, geopolymer/benzoxazine, etc.) is the other way to improve the hit resistance. However, in this case FST properties of the resulting composite should be strictly supervised.

4.2. Eco-fibres

A potential way to reduce environmental footprint of classic glass fibre reinforced phenol formaldehyde resin as used today in aviation linings is the substitution of glass fibres by natural fibres and/or recycled carbon fibres. As geopolymer matrix is showing increased fire resistivity to classic thermoset resin systems, a combination of the so-called eco-fibres with geopolymer matrix has been assessed for their potential in the second test batch of WP7.2.

Samples reinforced with rCF show the lowest smoke density and toxicity, followed by the reference of GF-PF. The high potential of geopolymer resin to reduce fumes and toxicity in cabin environment is therefore

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

shown. The mechanical properties of rCF combined with geopolymer show a promising potential, though a weak fibre matrix adhesion prevents better results.

A different picture is shown by the combination of flax fabric with geopolymer. While flammability, smoke density and toxicity tests show promising behaviour, the heat release is still too high for application in aviation interior. Furthermore, the mechanical properties assessed by flexural test are too low.

A hybrid with outer layers of rCF and an inner layer of flax shows good FST+HR results. Mechanical tests are not finished and will be added later in the final report.

Generally, the weak fibre-matrix adhesion is a challenge for the use of geopolymers. Cold plasma treatment of the fibres could be a solution. Furthermore, the influence of moisture must be carefully considered. Flax fibres are very absorbent and water has a strong effect on their modulus. Geopolymers are partly water based system. Flexural tests with pre-dried specimens are recommended to assess possible differences. GPL resin features a strong basic pH (>11) until it hardens which takes normally few hours until the pH factor of cured resin drops to neutral value. The effect on the flax by alkalinity should be observed in detail. Otherwise, a NaOH treatment with pH of about 13 is a standard process to modify natural fibres.

The calculated pore content of the rCF nonwoven reinforced geopolymer is up to 33% and needs to be better controlled in future manufacturing trials in order to get a better picture of the performance potential. The embedding in a fire resistant geopolymer matrix alone is not enough to protect the fire sensitive flax fibres. It has to be explored, if a better composite quality with reduced void content is able to improve the heat release results considerably. If not, the addition of a flame retardant is needed to fulfil the demanding aviation requirements.

A possible way to enhance mechanical and fire properties could be a hybrid composite. The example of two rCF outer layers and one flax inner-layer shows comparable results to the reference GF-PF. Generally, for application in aviation interior linings, the highest attention should be given to the reduction of the panel weight by a lower density. This is the most effective way to reduce the environmental footprint by lowering the kerosene consumption during the use-phase. A Life Cycle Assessment (LCA) is recommended to compare possible variants with the state-of-the-art.

4.3. Fibre Metal Laminates

The second test batch consists of further tests for characterization of FML material properties. The tests contained DMA measurements as well as the determination of temperature dependent shear and compression properties. All Tests were conducted for FML and the used unidirectional prepreg material that was used for FML processing. The results show dropping properties for strength and shear properties with moderate amount until 150°C and higher degradation above 150°C. The compression modulus in fibre direction is almost not influenced until 150°C. Further results with discussion will be included in the final report (D7.13).

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.

Moreover, the second batch includes fire tests with simultaneous mechanical loading. To this, a test stand was developed. The test stand allows axial compression loading of curved specimens that are potted within concrete-filled moulds. The moulds are mounted to a press. Within the test, the specimens are preloaded by 50MPa axial compression load. The test stand construction includes additional insulation and allows fire loading to the specimen while the compressive force is still loaded. Multiple tests have been conducted on FML specimens showing the pillow effect that works as insulation to the rear laminate plies. Additional to the burn-through resistance of FMLs, the structural integrity was investigated with respect to such a fire scenario. The collapse of the structure was investigated to be after sideway cracking of the developed pillows. As a consequence to this, temperature rises at the rear plies causing locally decreasing mechanical properties resulting in structural collapse.

Future Sky Safety has received funding from the EU's Horizon 2020 Research and Innovation Programme, under Grant Agreement No. 640597.