Improving material solutions to mitigate fire, smoke and fumes in cabin environment – final report

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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 Project P7 "Mitigating Risks of Fire, Smoke and Fumes" of Future Sky Safety, and is a synthesis report of the work performed within the Work Package 7.2 "Improving material solutions to mitigate fire, smoke and fumes in cabin environment (plus toxicity)" during the project.

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Acronyms

Acronym	Definition
AITM	Airbus Industries Test Method
ASTM	American Society for Testing and Materials
BVID	Barely Visible Impact Damage
CD	Cross Direction
CFRGC	Carbon Fiber Reinforced Geopolymer Composite
CFRP	Carbon fibre reinforced plastic
CIT4min	Conventional Toxicity Index In The 4th Minute (Dimensionless)
CIT8min	Conventional Toxicity Index In The 8th Minute (Dimensionless)
CO	Carbon Monoxide
CO2	Carbon Dioxide
CTE	Coefficient of thermal expansion
Dc	Specific Optical Density Of Smoke After The Measurement
DLR	Deutschen Zentrums für Luft- und Raumfahrt
DMA	Dynamic mechanical analysis
Ds10	Specific Optical Density Of Smoke In The 10th Minute (Dimensionless)
Ds4	Specific Optical Density Of Smoke In The 4th Minute (Dimensionless)
DSC	Differential Scanning Calorimetry
Ds_{max}	Maximal Specific Optical Density Of Smoke (Dimensionless)
F	Flammability / Flame Propagation
FAA	Federal Aviation Administration
FED30min	Fractional Effective Dose (Total, Dimensionless)
FML	Fibre metal laminate
FRP	Fibre reinforced plastic
FST	Fire/Smoke/Toxicity
FST	Fire smoke toxicity
GF	Glass Fibre
GFRP	Glass fibre reinforced plastic
GP / GPL	Geopolymer
Gr	Gradient

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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
RTM	Resin transfer moulding
S	Smoke
SD	Smoke Density
T	Toxicity
TGA	Thermogravimetric analysis
TMA	Thermomechanical analysis
vCF	Virgin Carbon Fibre
VID	Visible Impact Damage
VOF4	Accumulated Value Of Specific Optical Density Of Smoke In The First 4 Minutes
VZLU	Czech Aerospace Research Centre

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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 Fibre 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

The overall objective of WP7.2 "Improving material solutions to mitigate fire, smoke and fumes in cabin environment (plus toxicity)" is to investigate the potential of materials that may contribute to reduce the impact of fire and smoke in the cabin environment. Specific avenues that are being investigated include:

- Definition of tests to characterize material properties with respect to their fire and mechanical properties,
- Manufacturing of samples from composite materials with promising ,
- Developing and characterizing of new materials and their combinations for an improved fire behaviour of interior and structural materials,
- Model material degradation with respect to fire, fumes and smoke risks in the cabin environment.

The objective of this deliverable (D7.13) is to provide a summary of the results obtained by WP7.2 of Future Sky Safety. Detailed results of the work performed are documented in the following deliverables:

- 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 [1].
- D7.8 is a summary of results from the second batch of tests in WP7.2 of the FSS project [2].
- D7.10 reports the results of modelling and simulation [3].

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Results & Conclusions

Geopolymers (VZLU)

Geopolymers are amorphous aluminosilicate materials that combine low temperature, polymer-like processing with high temperature stability. This combination of properties makes geopolymers an interesting alternative to existing 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 range.

Geopolymers feature high temperature stability and fire resistibility, achieved by low temperature processing, limited generation of toxic fumes and smokes, low thermal conductivity, good specific strength and low price.

On the other hand, geopolymer matrix is relatively brittle and its specific gravity is higher than that of organic resins. Due to high alkalinity of uncured resin, range of applicable reinforcement phases is limited to alkali-resistant fibers. Carbon (aramid alternatively) reinforcement was chosen as the optimal option.

In the frames of the project, Carbon Fiber Reinforced Geopolymer Composites (CFRGC) were subject of research in undermentioned spheres:

- Flame resistance,
- Fire smoke toxicity properties,
- Mechanical properties (including environmental expositions and impact tests).

Particular interest was paid to development of geopolymer based hard foam as a replacement of state of the art organic core materials.

In following paragraphs short summaries of project results are stated:

<u>Summary flame resistance:</u>

Monolithic test panels:

The panels were subjected to flame penetration test per CS25, App.F, Part III. During the tests, there was no evidence of flame penetration or smoke generation in case of CFRGC panels. The panels showed very good flame resistance and after-test structural integrity. No mechanical damage or overt smoke generation were registered. Referential glass / phenolic panels typically generated smoke at the beginning of the test which was followed by short flash out on the back side of the panel. Glass / phenolic panels lost their mechanical integrity due to resin burnout. Vertical test per CS25, App.F, Part I: Both CFRGC and glass / phenol panels passed the test. Longer burn length and smoke generation were registered at glass / phenolic specimens.

Sandwich test panels:

During the tests per CS25, App. F, Part III, there was no evidence of any flame penetration in case of the panels provided with CFRGC skins. Strong blow up ("pillow") effect was registered both on foam and honeycomb core CFRGC panels. This was partly eliminated by applying metal frame on the panel edge. No other damages were detected. CFRGC sandwich panels showed very good after-test structural integrity.

Referential glass / phenolic test panels typically have blown-up resulting in edge rip and leakage of smoke from burning core. Large areas of burned out resin were evident on the flame exposed sides of the panels (spots of bare fabric). The panels lost their mechanical integrity due to resin burnout.

Fire smoke toxicity properties:

From the point of view of criteria under review, CFRGC gave significantly better results in comparison with referential glass/phenol in all evaluated FST parameters (conventional toxicity indexes, CO and CO2 concentrations, specific optical density of smoke, accumulated value of specific optical density of smoke and fractional effective dose).

Static strengths and modules

Static strengths and modules (tensile, flexural, shear, compression, ILSS) of CFRGC composite material were evaluated without and with environmental expositions applied. The most significant drop of properties was registered after “hot-wet 1” and “salt mist” expositions (these of 100 % humidity), especially in case of shear tested specimens. Influence of working fluids (fuel, hydraulic oil, lubricant) were found to be only moderate. Generally, CFRGC mechanical properties were found to be about the same as these of referential glass/phenol.

Drum peel tests

Drum peel tests per ASTM D1781 - 98 of sandwich panels constructed of CFRGC skins and honeycomb / foam core were carried out. Three types of inorganic and two types of organic adhesives were tested. As a referential group of specimens, GURIT PHG 600 glass/phenolic prepreg based sandwiches were employed. In the group of foam core specimens the best results showed GPL30 (geopolymer resin) bonded specimens. In the group of honeycomb core specimens the best results showed Resbond® 989 (ceramic adhesive) bonded specimens. Generally, foam core specimens provided better peel strengths.

Impacts

In the 1st batch impact tests of sandwich panels constructed of foam core and a) carbon fiber / CFRGC skins and b) carbon fiber / geopolymer / phenol hybrid skins were conducted. Visible Impact Damage (VID) and Barely Visible Impact Damage (BVID) were evaluated with and without previous hot-wet conditioning. Comparison of both a) and b) types of specimens to referential standard materials showed worse impact resistance of both geopolymers and hybrids.

In the 2nd test batch methodology changes were realized:

- monolithic test specimens were utilized (simplified tests evaluation),
- physical surface treatment of fibers was applied (better wettability by geopolymer was proved),
- ductile aramid fibers were incorporated to composite lay-up (premise of impact resistance improvement).

The test results of the 2nd batch showed clear asset of aramid fibers incorporation into composite lay-up (resulted in as many as six times smaller impact depths). Hybrid carbon/aramid geopolymer also proved the highest energy absorb capability and the lowest specific weight from compared materials.

Geopolymer foam

Hard structural geopolymer foam was developed as potential replacement of state of the art organic sandwich cores. Testing of the first batch of the foam showed, as expected, excellent fire resistivity and almost zero generation of smoke and combustion gases even at long term exposition at 1350^{o}C. At the other side the 1st batch specimens feature higher specific weight-to-strength ratio and brittleness compared to thermoplastic foams.

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Flax fibres and recycled carbon fibres (DLR)

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. As the interest in using ecologically improved materials in aviation is high, it is very important to assess their impact on fire properties and the air quality in cabin environment.

During the first batch of tests, flax and recycled carbon fibres have been used to manufacture non-woven in the DLR laboratory. A promising bio-based resin system (furan) that exhibits comparable characteristics to state-of-the-art phenolic resin was used as matrix for the composites. Fire and mechanical tests show the potential and challenges of the different material combinations. When using 100% recycled carbon fibres as reinforcement, the fire and mechanical properties are very promising, while the heat release is still too high to fulfil the demanding aviation requirements. The application of flax fibres in a hybrid with rCF or as sole reinforcement leads to considerable challenges to fulfil the fire requirements. Therefore the additional use of fire retardants is a mandatory. Resin additives, fibre sizing and coatings need to be tested for the effectiveness and impact on mechanical properties, air quality and weight of the interior panel.

Flax fabric (plain weave) and a nonwoven from rCF were 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 embedded in 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.

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-PFpanels. 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.

Fibre-Metal-Laminates (DLR)

Fibre metal laminates (FML) were investigated to improve the present CFRP material behavior within a fire scenario. To this, it was shown, that the metal layers acting as gas barrier significantly reduce the generation of smoke and toxic gasses. Moreover, the burn through resistance is improved allowing increased duration of mechanical performance of the structure. To further investigate this, the compression under fire exposure test was developed.

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Future work is needed to further improve the revealed possible effects. This includes for instance layup changes also leading to reduced weight of the laminates. Furthermore, hygroscopic effects or the effect of impacts to the behavior within a fire scenario must be studied.

Compression under Fire Exposure Test (DLR)

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. The tested specimens have a dimension of 200mm length, a 120mm width and a radius of 245mm. 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 structural collapse due to stability (buckling).

Five specimens were tested, four of them with an aperture and one without. The specimens with the aperture showed a “pillow-effect”, insulating layers of gases within the laminate.

The tests performed so far show the functionality of the CuFex test-stand. Now it is possible to compare different materials under fire exposure and to evaluate their fire resistance. This can be done with relatively small and simple specimens which allows to perform numerous test in a short time-span at low costs.

In the future a direct comparison of the used FML to the reference-material has to be done to quantify the pillow-effect and to get a deeper understanding of the processes of decomposition. Also the number of steel-layers should be changed to determine how many layers are needed to generate an efficient barrier for decomposition and burn-gases. Furthermore, the test could be extended by specimens with stiffeners on the back side. Such test would deliver further understanding of the insulating effect and how the mechanical performance degrades on a structural level.

Modelling and Simulation (LEONARDO, DLR)

Test results obtained during the test campaign allowed collecting all the data necessary to improve the FlamePTM characterizing in depth the test specimen material, in order to simulate and study the specimen behavior in terms of:

- Material degradation: gas produced by the pyrolysis phenomena,
- Specimen structural behavior: deformation and stress.

Structural analysis results have been not reported in the deliverable due to the negligible deformation detected with the model. It is due to the fact that the flame penetration test is a static test and the only structural effect is due to the effect of the gas produced in the specimen. It has been not considered yet in the model.

FlamePTM with the improvement described in this deliverable assures a depth simulation of flame penetration test with the following reported important benefit for aircraft companies:

- Reduce the time and costs of specimen suppling,Reduced test time, the experimental activity is minimized to the confirmation of the results for the design approval,
- Reduce number of development tests and certification tests (cost reduction),

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- Reduce risk associated to the development phase: the refinement is anticipated in the concept phase (cost reduction),
- A wide spectrum of configurations and cases (optimized design).

The simulation model to investigate FML behaviour within the CuFex facility uses temperature and state dependent material properties. The insulating pillow effect of the FML could be reproduced by the simulation and the drop of mechanical performance due to the decomposing matrix was studied. Nevertheless, several simplifications were assumed within the FE model and have to be studied within future work to enhance the model.

The simulation of the combined mechanical and fire loading supports the understanding of new material solutions. Further enhancements of the simulation strategy could lead to identify further improved material solutions such as optimized layups. Furthermore, this leads to a decreased effort of expensive testing.

Furthermore, a modelling strategy has been developed by DLR to investigate the structural behavior of FML with respect to simultaneous fire and mechanical loading. To decrease computational effort, the simulation was conducted by sequential heat transfer and mechanical analysis. The material models used describe the temperature dependent material behavior of thermal and mechanical properties. They are based on measurements and several assumptions. However, the comparisons of simulation and CuFex-experiment show reasonable agreement. The present simulation strategy of DLR is a good basis for future developments and enhancements such as further investigation of material properties. Moreover, within future work, the simulation is intended to be enhanced for a thermos-mechanically coupled analysis leading to the determination of improved FML layups with respect to combined fire and mechanical loading.

Applicability

Geopolymers

Natural nonflammability is an inherent property of the geopolymers and is not achieved by additives or by mechanical means, as in case of the state of the art reactoplastics and thermoplastics. Extensive tests, carried out in the frames of WP7.2 proved unbeatable resistance to fire and outstanding FST properties of carbon fiber-geopolymer composite material. Mechanical properties of the material comparable to the present phenolic based glass reinforced materials were also demonstrated. Due to natural mechanical properties of geopolymer matrix the resulting composite features specific characteristics (reduced ductility) and so applications in primary aerospace structures is not contemplated. Secondary structures as internal linings in cargo or cabin areas, fire bulkheads or temperature exposed pipelines could be examples of suitable aircraft applications. Geopolymer foam that was also developed and tested in the project can be utilized as flame / heat insensitive core in sandwich structures. Fiber reinforced geopolymer is also ideal for effective making of high temperature molds in aerospace manufacturing. Also many applications in non-aerospace business are put up: e.g. interiors of ground vehicles, fireproof wood or concrete reinforcement or blades of high temperature axial fans (developed and tested in VZLU). The research and tests carried out in frames of the WP7.2 are just a preview of very complex issue. Presently most applications of geopolymers are in the civil engineering. So that many material characteristics typical for aircraft engineering haven‘t been studied yet. Many topics are left to explore (fatigue behaviour, properties during and after heat exposition…) to get a complete understanding of the material.

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Modelling

An aim of the FSS Project P7 has been the development of a material test results database available to validate the model developed by the Aeronautical industry in the frame of material behavior in fire condition. In the frame of WP7.2 test results collected in the database defined during the Project P7 have been used to improve and validate the Flame Penetration Test Model (FlamePTM) developed by Leonardo. Improvement and validation of the FlamePTM addresses key industrial problems with low degree of confidence that need presently extensive experimental verification. Activities has contributed to advancing physical understanding of multi-physics phenomena of the material degradation in fire condition. In aeronautical industry field have a Flame penetration test model validated on the basis of test results that allow to perform in virtually way the test required by the certification rules allow to:

- reduce risk associated to the development phase: the refinement is anticipated in the concept phase. With consequently man power and cost of test reduction.
- Reduce the time and costs of specimen suppling.
- Reduce test time, the experimental activity is minimized to the confirmation of the results for the design approval.
- Reduce number of development tests and certification tests (cost reduction)
- Have the possibility to use the model to study a wide spectrum of configurations and cases in order to optimize the project.

FML & CUFEX

It has been shown that Fibre-Metal-Laminates (FML) are capable of improving the FST behaviour compared to their CFRP base material. This could be used not only for aviation load-bearing structures but also for other means of transport with stringent fire requirements (e.g. railway, marine). A further improvement of the FML properties is expected by tailored laminate design (e.g. thinner metal sheets and their positioning) that might be derived by using and enhancing the modelling methods developed in FSS. Furthermore, possible multifunctional aspects of FML compared to standard CFRP should be investigated (e.g. electrical properties). A new test stand for axial compression loading under fire exposure (CUFEX) has been developed and build-up at DLR. First tests on FML show promising results. The CUFEX test stand can be used for future research activities and further understanding of scale effects. This could include stiffened specimens to facilitate the step from coupon level to a more realistic structure level.

Eco-Fibres

Bio-based fibres like flax have a great potential to substitute glass fibres in composite applications due to their good specific stiffness. However, their low strength and especially inferior fire properties compared to GFRP are an obstacle for the application in aviation secondary structures and interior panels. In FSS WP7.2 it was shown that a hybridization of flax fibres with recycled carbon fibres (rCF) can enhance the mechanical properties compared to standard natural fibre reinforced polymers (NFRP). The additional rCF are able to improve the FST properties if they are concentrated on the surface of the composites to isolate the natural fibres from the flame. The mixing ratio and fibre distribution has to be further understood and improved. Furthermore, the use of an intrinsically fire resistant matrix system like geopolymers has shown promising FST results utilizing rCF and flax fibres. Future investigations are necessary to improve the fibre matrix adhesion of geopolymers and flax/rCF fibres in order to improve the composite mechanical properties. A Life Cycle Assessment (LCA) is finally needed for a detailed characterization of different environmental impacts during the complete life cycle to compare the new eco-composites with currently used GFRP.

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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

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LIST OF TABLES

1 INTRODUCTION

1.1. The Programme

FUTURE SKY SAFETY is an EU-funded transport research programme in the field of European aviation safety, with an estimated initial budget of about € 30 million, bringing together 32 European partners to develop new tools and approaches to aviation safety. The first phase of the Programme research focuses on four main topics:

- Building ultra-resilient vehicles and improving the cabin safety,
- Reducing risk of accidents,
- Improving processes and technologies to achieve near-total control over the safety risks,
- Improving safety performance under unexpected circumstances.

The Programme will also help to coordinate the research and innovation agendas of several countries and institutions, as well as create synergies with other EU initiatives in the field (e.g. SESAR,). Future Sky Safety is set up with an expected duration of seven years, divided into two phases of which the first one of 4 years has been formally approved. The Programme started on the 1st of January 2015.

FUTURE SKY SAFETY contributes to the EC Work Programme Topic MG.1.4-2014 Coordinated research and innovation actions targeting the highest levels of safety for European aviation, in Call/Area Mobility for Growth – Aviation of Horizon 2020 Societal Challenge Smart, Green and Integrated Transport. FUTURE SKY SAFETY addresses the Safety challenges of the ACARE Strategic Research and Innovation Agenda (SRIA).

1.2. Project context

Recent studies] have shown that, though “fires in flights” as a direct cause represented only 5% of fatalities, “fire/smoke resulting from impact” accounted for 36% of all fatal accidents. Often aircraft occupants have survived the impact only to be incapacitated by toxic fumes and/or heat, e.g. temperatures can rise above 600-700°C after only three minutes. Toxic fumes originate from components such as aviation fuel and combustible materials, producing various gases dependent on the composition of the material.

In recent years the development of more lightweight aircraft has seen an increased use of composite materials in primary structures, e.g. fuselages, as well as secondary and interior structures, such as furnishings. These materials have desirable properties such as corrosion resistance and high strength, however from a safety point of view the use of these materials may require specific controls concerning their behaviour when exposed to fire, or during normal conditions. The project seeks to address this safety aspect within three work packages:

- WP7.1 – The first work package aims to test and thus improve understanding of the effects of fire on these materials,
- WP7.2 – The second work package aims to develop and propose improved material solutions to mitigate fire, smoke and fume,
- WP7.3 – The third work package, for which this report is the final deliverable, aims to investigate the possible effects of new materials and technologies on the on-board air quality with the objective to further improve the air quality.

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1.3. Work package content

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.

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 using natural fibres and (recycled) man-made fibres is to substitute classic cabin materials (glass fibre fabric) for reduced environmental impacts. 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 fibres 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 overall objective of WP7.2 is to investigate the potential of materials that may contribute to reduce the impact of fire and smoke in the cabin environment. Specific avenues to be investigated include:

- Definition of tests to characterize material properties with respect to their fire and mechanical properties,
- Manufacturing of samples from composite materials with promising ,
- Developing and characterizing of new materials and their combinations for an improved fire behaviour of interior and structural materials,
- Model material degradation with respect to fire, fumes and smoke risks in the cabin environment.

The objective of this deliverable is to provide a summary of the results obtained by WP7.2.

1.5. Approach

Geopolymers

For 'non-flammable' shall be considered such substances that does not burn, smolder or carbonificate at normal pressure by the action of fire or high temperature. It is known that most inorganic substances meet these conditions. Thus application of inorganic based fibre composites is the possible way to meet the WP7.2 tasks. If appropriate lightweight, fire-resistant (e.g. carbon) fibres are applied in conjunction with inherently non-combustible inorganic matrix, there is an opportunity to get the material featuring new level of fire and FST safety. A promising candidate for such materials, are the geopolymer compounds. Geopolymers are a class of totally inorganic, alumino-silicate based ceramics. They are rigid gels, which are made under relatively ambient conditions of temperature and pressure into crystalline or glass-ceramic materials [6].

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In WP7.2 Carbon Fibre Reinforced Geopolymer Composites (CFRGC) were subject of research in undermentioned spheres:

- Flame resistance,
- Fire smoke toxicity properties,
- Mechanical properties (including environmental expositions and impact tests).

Particular interest was paid to development of geopolymer based hard foam as a replacement of state of the art organic core materials.

Eco-Reinforcements

As the reduction of ecological impacts is getting more and more attention as important factor for future transport activities, it is another approach of P7.2 in Future Sky Safety to assess the potential of ecological improved composite constituents for their effect on fire, smoke and toxicity. At DLR, semi-finished products from natural fibres (flax) and recycled carbon fibres in combination with different matrix systems have been tested. Starting with a bio-based furan resin, the second step was to use the flame resistant geopolymer matrix together with partner VZLU. Exploitation of polymers matrices in combination with standard and renewable/recycled composite materials could pave the way to acceptable overall mechanical properties of geopolymers material systems. The characterization tests used for the eco-reinforcements with furan and geopolymer resin were simple mechanical tests (flexural) and different fire tests from aviation standards (Flammability, Heat Release, Smoke & Toxicity).

FML

Compared to common CFRP materials, Fibre-Metal-Laminates (FML) propose improved behaviour under fire exposure. This behaviour is produced by steel layers that are integrated into the laminate and act as gas barrier. To investigate and underline this, subject of research were smoke toxicity and smoke density tests, as well as burn through tests with respect to varying layups. Furthermore, a new test facility was developed to investigate the mechanical performance of FML while it is exposed to fire. Within the test specimens that are exposed to axial compression are simultaneously loaded to fire. The test is intended to study the insulation effects, how material degradation proceeds and if the material can still be expected as load carrying in such a scenario. Since manufacturing and testing is a huge effort a simulation method is developed. The aim of the simulation is to decrease future testing effort and to detailed investigate mechanical degradation proceeding of FML within a fire scenario. To this, material properties are determined with respect to their temperature dependency. The material characterization does also lead to further knowledge about FML materials.

1.6. Structure of the document

The document is adopting the Future Sky Safety template and general structure. As a whole, this document is a synthesis report summarizing the results achieved throughout the project in WP7.2. Chapter 1 covers an introduction summarizing the program content and the P7 research objectives and approach. A short summary of the tests standards is presented in Chapter 2 In Chapters 3, 4and 5 the proposed innovative materials solutions are described and the main test results presented. Chapter 6 gives an overview of a novel compression under fire exposure test developed at DLR. Chapter 7 will present results of modelling and simulation, while chapter 8 provides conclusions and recommendations.

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

2 TEST STANDARDS

This study provides the requirements and specifications of the tests and studied proposed highly resistant materials. 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. The material tests are described considering the test methods, the specimen size, and the realisation. Test requirements have been defined for interiors panels (lining), in terms of certification rules and relative test in compliance with them, and the definition of material characteristics that must be measured during the test useful to the numerical correlation of numerical model. The present results of actions taken during preparation of this study are related to definition of suitable and innovative material systems with high potential to mitigate and protect from fire, smoke and fumes in cabin environment while respecting industrial safety requirements and usage of state-of-the art simulation tools. A summary of planned experiments is shown in Table 1.

Table 1: Initial plan of experiments

Test	*Test method*	*Parameter*
Flame Propagation / Flammability	AITM 2.0002A (vertical, 60 sec)	Fire properties
Flame Penetration Resistance	CS 25 Appendix F, part III	Fire properties
Heat Release Rate	AITM 2.0006	Fire properties
Smoke Density	AITM 2.0007A	Fire properties
Smoke Toxicity	AITM 3.0005	Fire properties
Back surface temperature profile	DLR test method	Fire properties
Mechanical properties after fire exposure	DLR test method	Fire properties
Tension	ISO 527-4 ASTM D 3039	Mechanical properties
Compression	PrEN 2850 ASTM D 6641	Mechanical properties
Flexural (3PB)	ISO 178	Mechanical properties
Lap shear	DIN EN 6031	Mechanical properties
Impact (CAI)	AITM 1.0010 ASTM D 7137	Mechanical properties
Shear	DIN EN 6031	Mechanical properties
DMA	ISO 6721, Part 3	Mechanical/thermal properties
TMA	DIN 57752	Mechanical/thermal properties
DSC	VZLU test method	Mechanical/thermal properties
TGA	VZLU test method	Thermal properties
Density	Hydrostatic weighting	Material value

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On the basis of the requirements and tests defined, the tests have been executed. Aim of the tests was the verification of the compliance with the certification requirements, measurement of the material characteristics and evaluation of the material capacity to withstand at high temperature/fire condition.

The given material characteristics measured are input data for activity of experimental/numerical correlation of the simulation model for chosen material solutions like fibre-metal-laminates (FML).

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3 GEOPOLYMERS

The following subchapters give information about properties of carbon fibre reinforced geopolymer composites (CFRGC) and geopolymer based foam as they were found out in the frames of the project. Undermentioned spheres were studied:

- Flame penetration resistance,
- Fire smoke toxicity properties,
- Mechanical properties,
- Geopolymer foams.

General description of manufacturing procedures of both geopolymer and referential glass/phenol test panels are stated in the report FSS_P7_VZLU_D7.5 [1].

3.1. Flame Resistance

Monolithic panels

CFRGC monolithic panels were subjected to tests according to standards:

- Flame penetration test per CS-25, App. F, Part III,
- Vertical test per CS-25, App. F, Part I.

Flame penetration test per CS-25, App. F, Part III

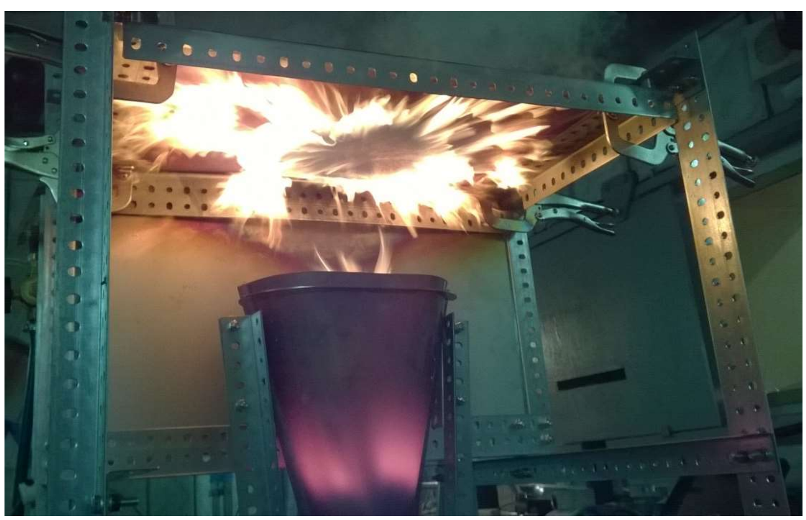

Figure 1: Flame penetration test arrangement

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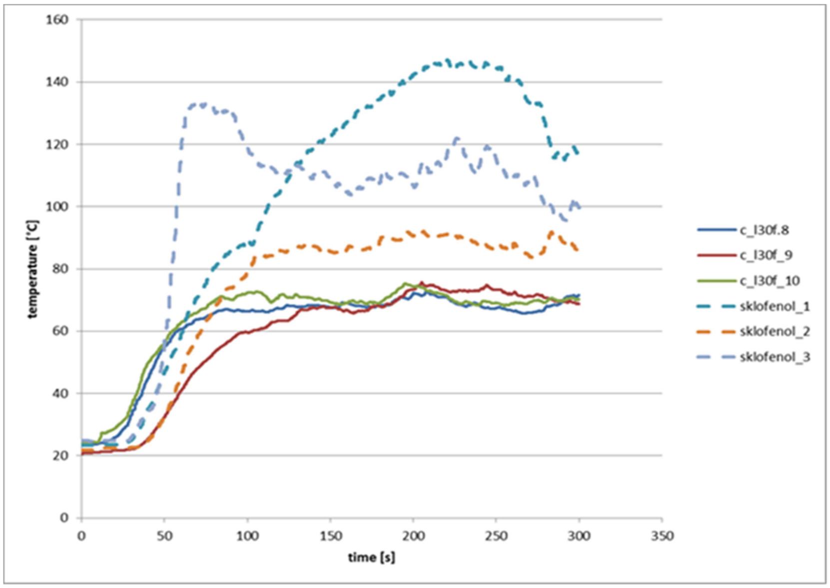

Figure 2: Temperatures 101 mm above the panels: CFRGC (full lines) and referential glass/phenol (dashed lines). Limit temperature per CS25, App. F: 204°C.

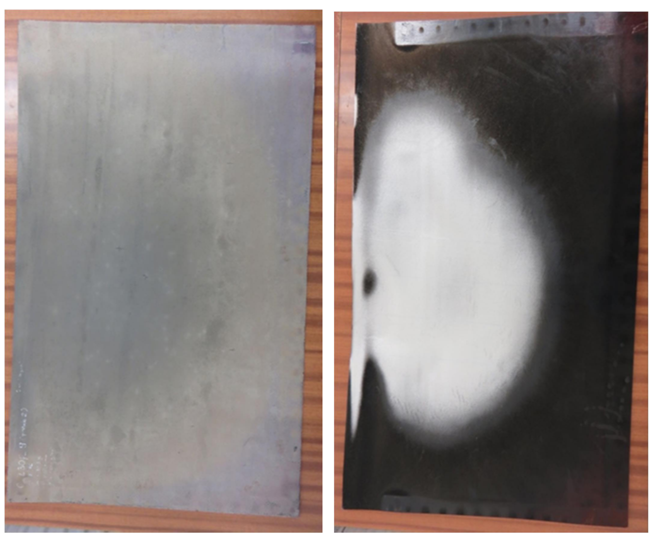

Figure 3: CFRGC panel (left) after the test (exposed side) and referential glass/phenolic panel (right) after the test (exposed side). White spot is bare fabric where phenolic resin has completely burned off.

Vertical test per CS-25, App. F, Part I

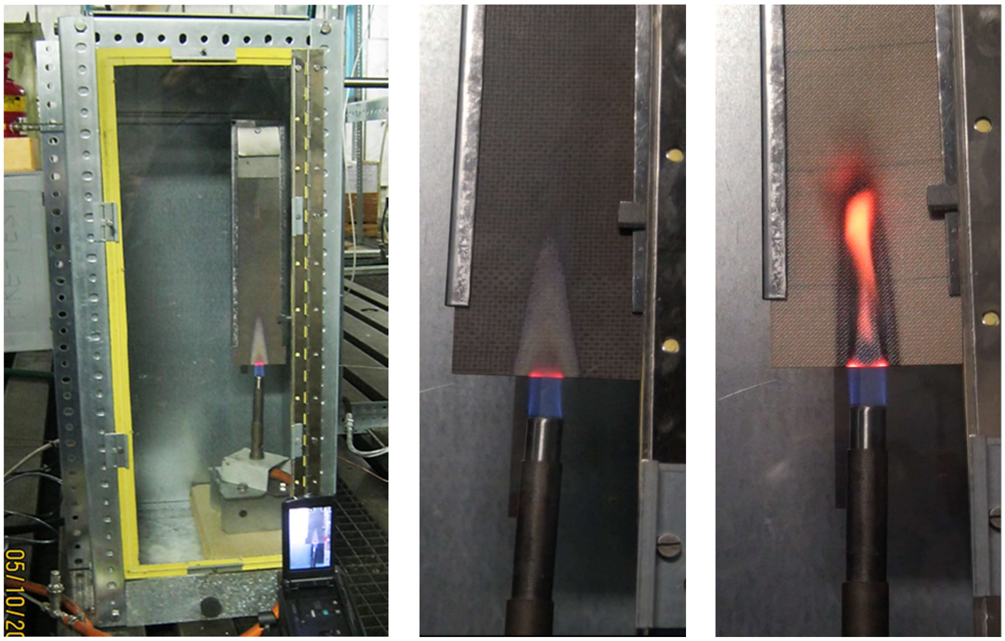

Figure 4: Test chamber for vertical test (left). Panels in approx. 30th second of the test (CFRGC middle, glass/phenolic right)

Table 2: Evaluation of the vertical test per CS-25, App. F, Part I

	CFRGC	glass/phenolic
Flame time	0	0
Burn length	0	~ 1 cm
Flaming time of drippings	0	0

Sandwich panels

CFRGC sandwich panels were subjected to flame penetration test per CS-25, App. F, Part III. Panels featuring both honeycomb and foam cores were tested. Geometrical scheme of the panels and arrangement of the test are stated in the report FSS_P7_DLR_D7.8 [2].

Figure 5: CFRGC sandwich panel (honeycomb core) after the test

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Figure 6: Glass / phenolic sandwich panel (honeycomb core) after the test

Summary flame resistance

Monolithic panels:

During the flame penetration tests, there was no evidence of flame penetration or smoke generation in case of CFRGC panels. Geopolymer panels showed very good flame resistance and after test structural integrity. No mechanical damage or overt smoke generation were registered.

Referential glass / phenolic panels typically generated smoke at the beginning of the test which was followed by short flash out on the panel back side in the first minute of the test. Areas of fully burned out resin were then evident as white spots of bare fabric. Glass / phenolic panels lost their mechanical integrity due to resin burnout.

Vertical test: Both CFRGC and glass / phenol panels passed the test. Longer burn length and smoke generation were registered at glass / phenolic specimens.

Sandwich panels:

During the tests, there was no evidence of any flame penetration in case of the panels provided with CFRGC skins. Apparent blow up ("pillow") effect was registered both on foam and honeycomb core CFRGC panels. This was partly eliminated by applying metal frame on the panel edge. No other damages were detected. CFRGC sandwich panels showed very good after-test structural integrity.

Referential glass / phenolic test panels typically have blown-up resulting in edge split and smoke leakage from burned core. Large areas of burned out resin were evident on the flame exposed sides of the panels (spots of bare fabric). The panels lost their mechanical integrity.

3.2. FST Properties

Fire/Smoke/Toxicity properties of CFRGC and referential E-glass/phenolic composites were evaluated according to standards:

- ABD 0031 & AITM 3.0005: Toxic Components on Combustion Products,
- CS 25, App. F, Part IV: Heat Release Rate and the Heat Release,
- CS 25, App. F, Part V: Smoke Density,
- ISO 5659-2: 2013 Plastics – Smoke generation – Part 2.

Detail information about FST properties of both CFRGC and referential E-glass/phenolic composites are stated in the report FSS_P7_DLR_D7.8 [2].

Toxic Components on Combustion Products acc. to ABD 0031 & AITM 3.0005

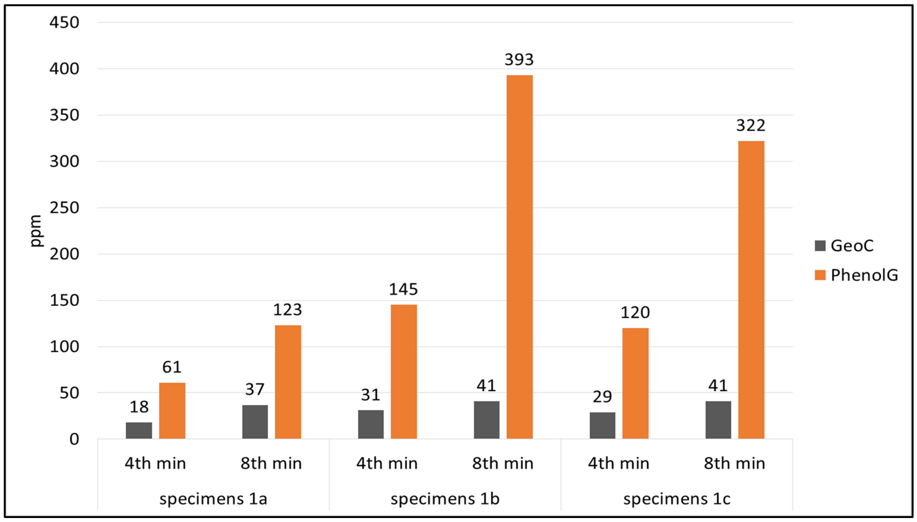

Figure 7: CO concentrations measured on 3 sets of specimens (values converted to standard pressure and temperature: 101.325 kPa, 25 °C)

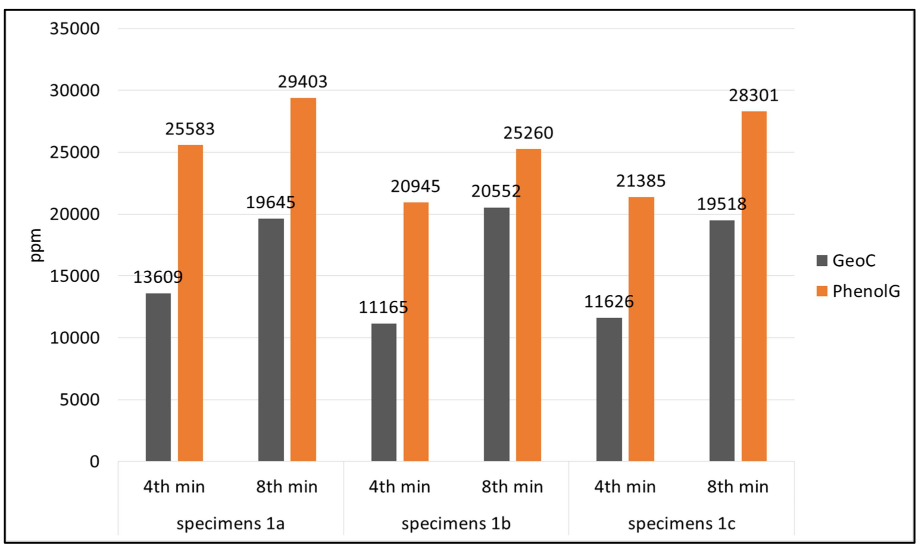

Figure 8: CO2 concentrations measured on 3 sets of specimens (values converted to standard pressure and temperature: 101.325 kPa, 25 °C)

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Smoke Density acc. to CS 25, App. F, Part V

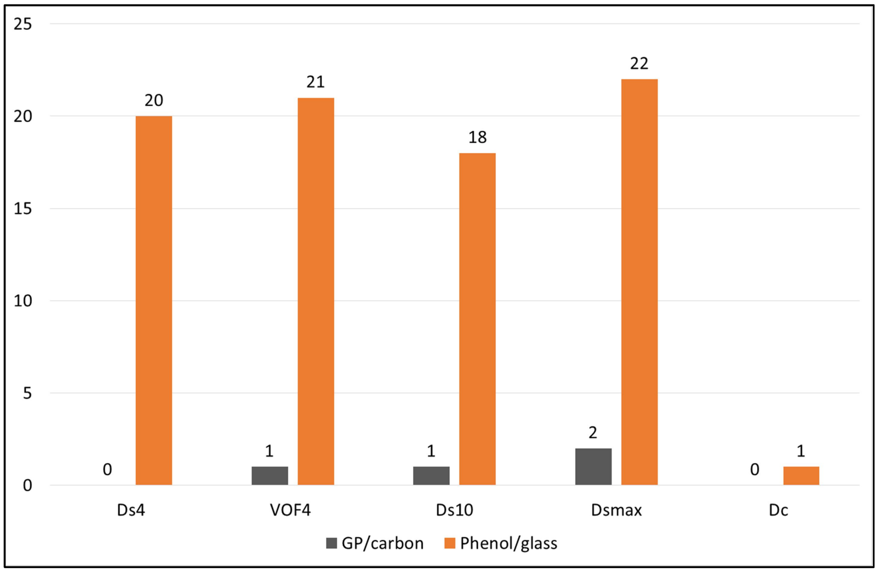

Figure 9: Smoke density test results

Heat Release Rate and Heat Release acc. to CS 25, APP. F, PART IV

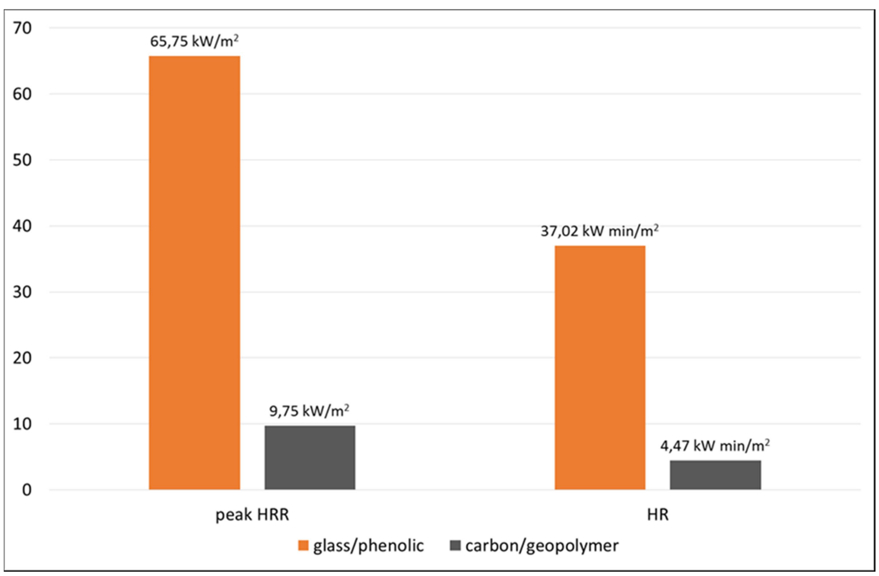

Figure 10: Heat release rate test results

Summary

From the point of view of criteria under review, CFRGC give significantly better results in comparison with referential glass/phenol in all evaluated FST parameters.

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3.3. Mechanical Properties

Following parameters of CFRGC were evaluated.

- static strengths and modules,
- peel resistance of sandwich skins,
- impact behaviour.

Selected sets of the test samples were submitted to environmental conditioning. Mechanical properties of CFRGC material were evaluated according to standards:

ASTM D3039	Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials
ASTM D6641	Standard Test Method for Compressive Properties of Polymer Matrix Composite Materials Using a Combined Loading Compression (CLC) Test Fixture
ČSN EN ISO 14125	Vlákny vyztužené plastové kompozity - Stanovení ohybových vlastností (Fiber Reinforced Polymer Composites – Determination of Flexural Properties)
ASTM D3518	Standard Test Method for In-Plane Shear Response of Polymer Matrix Composite Materials by Tensile Test of a ±45° Laminate
ASTM D2344	Standard Test Method for Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates
ASTM D1781 - 98	Standard Test Method for Climbing Drum Peel for Adhesives
ASTM D 7136/D 7136M – 07	Standard Test Method for Measuring the Damage Resistance of a Fiber-Reinforced Polymer Matrix Composite to a Drop-Weight Impact Event)
ASTM D 7137/D 7137M	Test Method for Compressive Residual Strength Properties of Damaged Polymer Matrix Composite Plates

Table 3: Mechanical properties - standards

Static strengths and modules

Applied materials:

For tensile, compression, flexural and ILSS tests 3K carbon fibers in form of the unidirectional tape were used (TCU 175 type, G. ANGELONI s.r.l). For shear tests, 12k fibers in form of ±45^{o} biaxial fabric were applied (ECC Carbon non-crimp fabric 200 g/m²).

Matrix: Geopolymer resin GPL30

Selected sets of the test samples were submitted to environmental conditioning:

- hot-wet 1: 70^{o}C tap water for 14 days (immersed) per EN ISO 175,
- hot-wet 2: 70^{o}C, 85% RH for 14 days per ČSN EN 60068-2-78,
- salt mist: 35^{o}C, 100% RH for 14 days per ČSN EN ISO 9227 NSS (NaCl concentration: 50g/l),
- working fluid 1: JET A-1 fuel (immersed) per EN ISO 175,
- working fluid 2: AeroShell Fluid 41 hydraulic oil (immersed) per EN ISO 175,
- working fluid 3: Mobil Jet Oil II aircraft gas turbine lubricant (immersed) per EN ISO 175.

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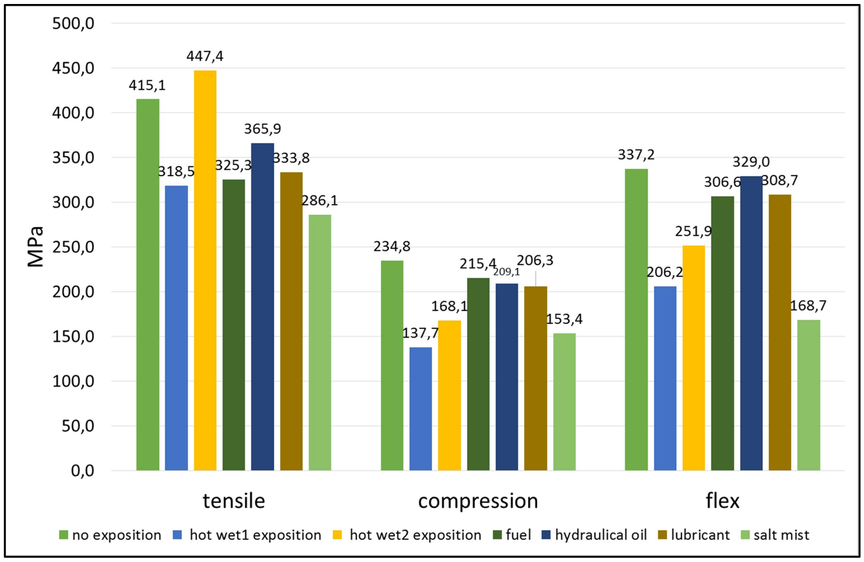

Figure 11: Static strengths - influence of environmental conditioning

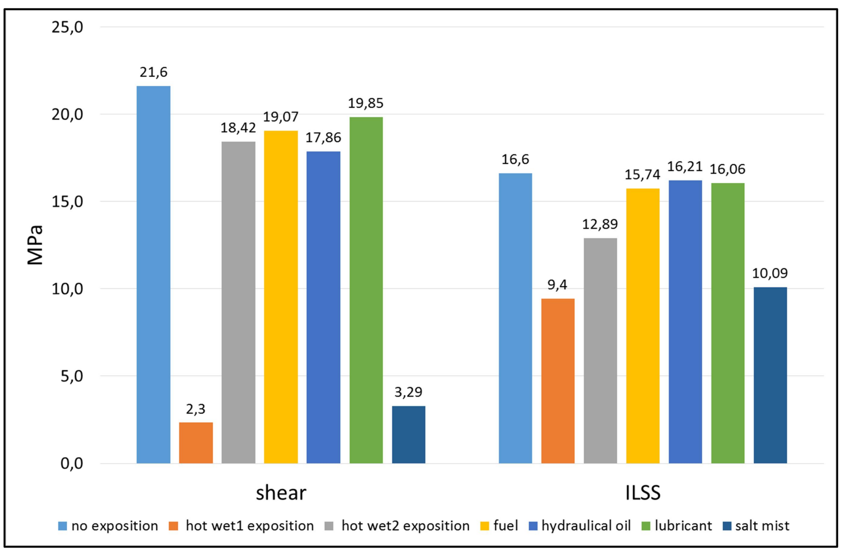

Figure 12: Static strengths - influence of environmental conditioning

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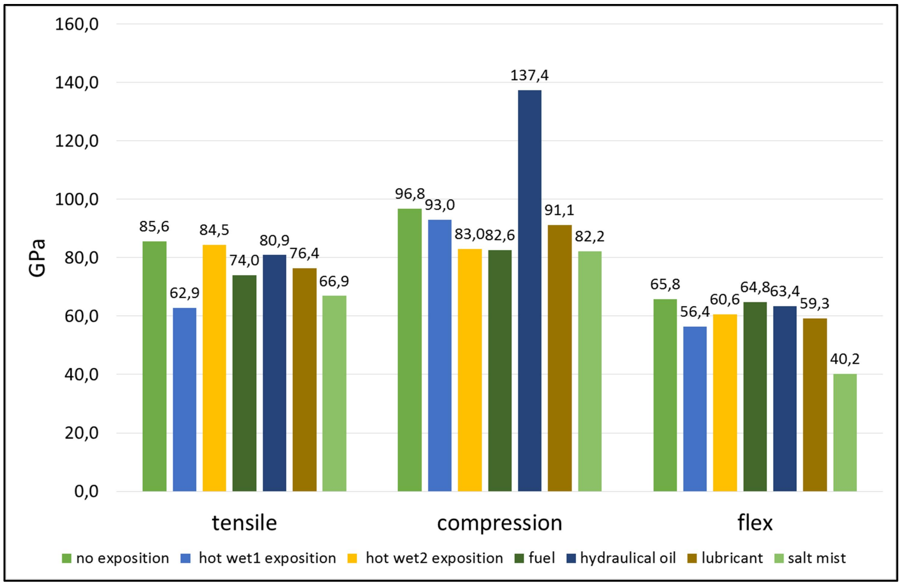

Figure 13: Modules - influence of environmental conditioning

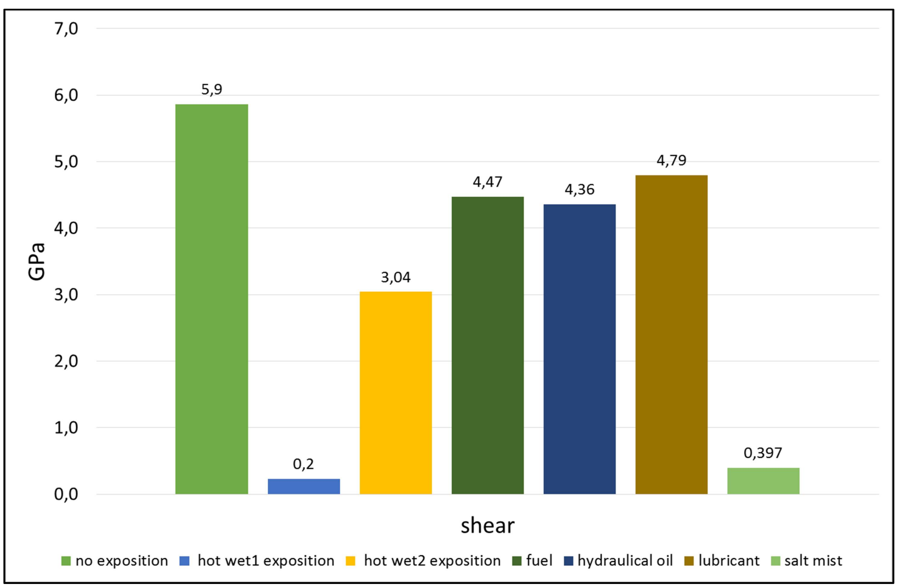

Figure 14: Modules - influence of environmental conditioning

Summary

Static strengths and modules of CFRGC composite material were evaluated without and with environmental expositions applied. The most significant drop of properties was registered after “hot-wet 1” and “salt mist” expositions (100 % humidity), especially in case of shear specimens. Influence of working fluids (fuel, hydraulic oil, lubricant) was found to be only moderate.

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Peel resistance of sandwich skins

Detail information about peel properties of both CFRGC and referential E-glass/phenolic sandwich structures are stated in the report FSS_P7_DLR_D7.8 [2].

Drum peel tests per ASTM D1781 - 98 of sandwich panels constructed of CFRGC skins and honeycomb / foam core were carried out. Several types of both organic and inorganic adhesives were tested. As a referential group of specimens, GURIT PHG 600-68-37 (style 7781) glass/phenolic prepreg based, co-cured sandwiches were employed. In group of foam core specimens the best results showed GPL30 laminating resin bonded specimens, closely followed by PH 600 prepreg bonded samples. In 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.

3.4. Impact behavior

1st batch of specimens (sandwiches)

Detail information about peel properties of both CFRGC and referential E-glass/phenolic sandwich structures are stated in the report FSS_P7_DLR_D7.8 [2].

2nd batch of specimens (monolithic laminates)

Tests methodology:

From experience of the 1st batch, in the 2nd batch following methodology changes were realized:

- monolithic test specimens were utilized,
- physical surface treatment of fibers was applied,
- ductile aramid fibers were incorporated to composite lay-up.

The 2nd batch impact tests were conceived as comparison of geopolymer based materials (carbon and hybrid carbon / aramid reinforced) with referential glass / phenolic prepreg laminate. The mass and the diameter of the impactor were stated as constants (3,738 kg, Ø16 mm). The tests were carried out at three energetic levels: 2J, 4J and 8J. It corresponds with drop heights of 55 mm, 110 mm and 219 mm. Three specimens of each material were tested at each energetic level. Impact (dent) depths and absorbed energy were evaluated. As the 1st batch, the tests were conducted on VZLU impact device S.U.P.R.

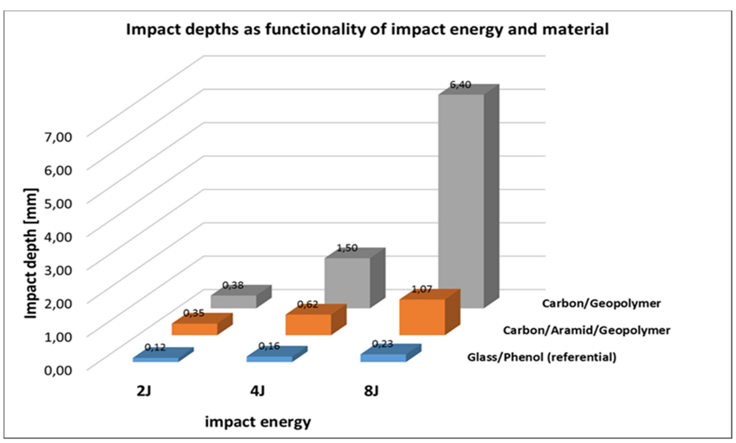

Figure 15: Impact depths as functionality of impact energy and material

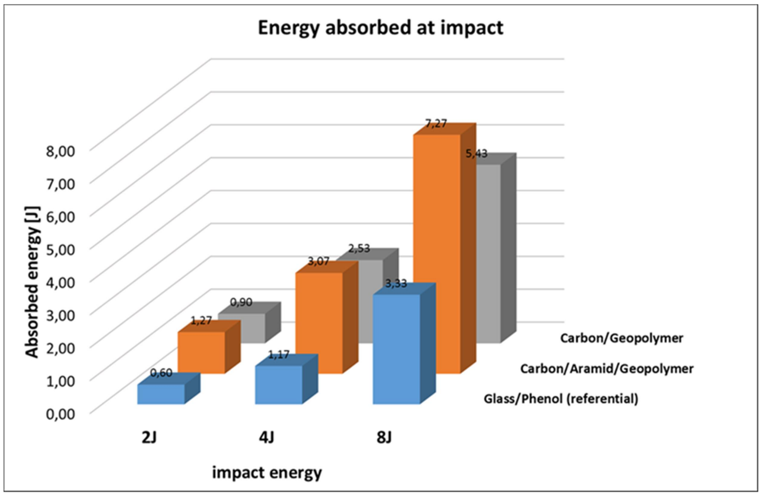

Figure 16: Energy absorbed by material as functionality of impact energy and material type

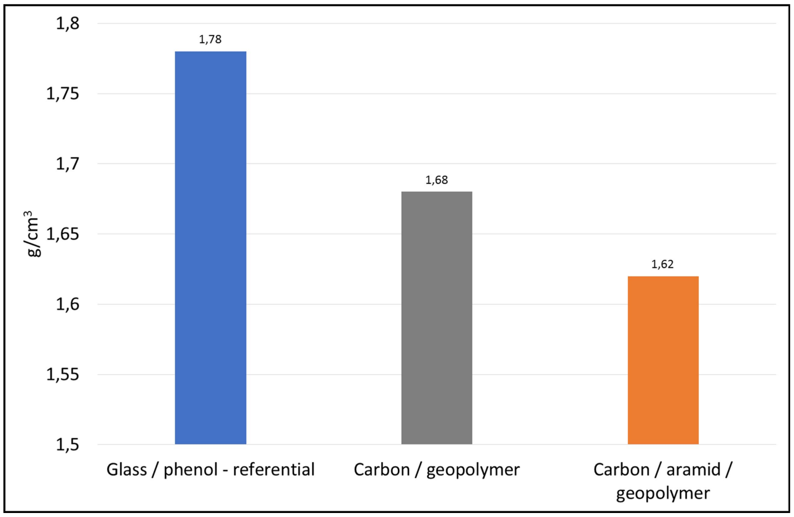

Figure 17: Comparison of specific weights of 2nd batch specimens

Summary

The test results of the 2nd batch showed clear asset of aramid fibers incorporation into composite lay-up. E.g. at 4 Joul level hybrid carbon/aramid geopolymer composite demonstrated 2,4x better impact resistance compared to carbon-only one and at 8 Joule level even six times better. Hybrid carbon/aramid geopolymer also proved the highest energy absorption capability and the lowest specific weight from compared materials.

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3.5. Geopolymer Foam

Mechanical properties of geopolymer foam were evaluated according to standards:

ASTM D1621	Standard Test Method for Compressive Properties of Rigid Cellular Plastics
ASTM D638-02a	Standard Test Method for Tensile Properties of Plastics
ISO 178	Plastics - Determination of flexural properties

Figure 18: Detail of the foam structure

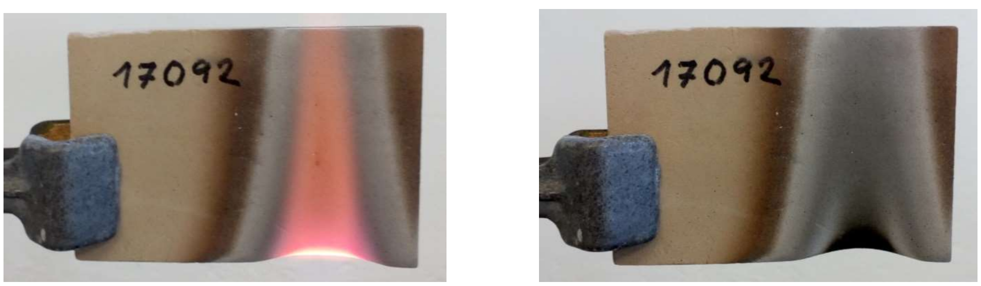

Figure 19: Foam specimen at ~1200°C (left, 5th minute of the test) and after 10 minutes at ~1200°C (right)

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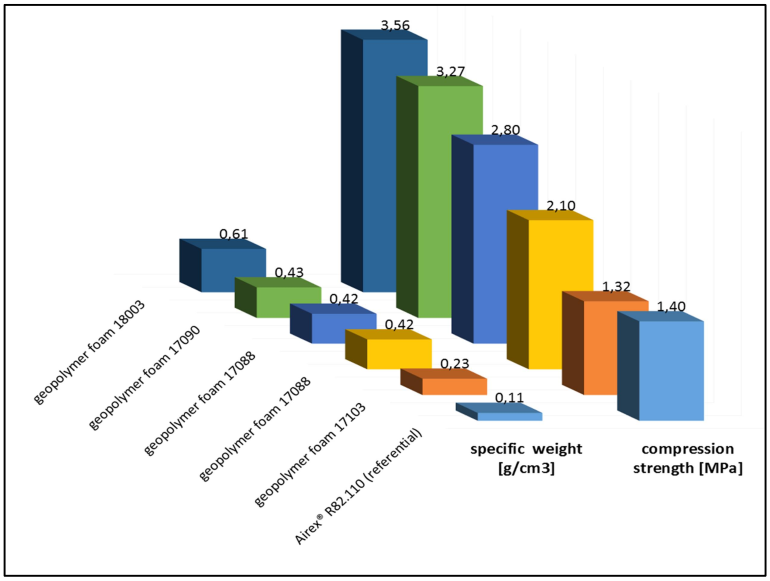

Figure 20: Compression strengths of the geopolymer foam of various specific weights. Compared to Airex© thermoplastic foam.

Summary

Hard structural geopolymer foam was developed as possible replacement of conventional sandwich cores. Flame penetration tests of the sandwich panels featuring standard organic foam or Nomex® cores showed adverse effects of expanding smoke and combustion gases to structural integrity of panels (blow-up effect, edge split). Testing of the first batch of the foam showed, as expected, excellent fire resistivity and almost zero generation of smoke or combustion gases. At the other side the 1st batch specimens feature higher specific weight-to-strength ratio and brittleness compared to thermoplastic cores.

4 ECO-REINFORCEMENTS (FLAX AND RECYCLED CARBON 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. A positive effect can be expected because the energy consumption to produce natural fibres is generally much lower compared to glass or especially carbon fibres. The same is expected for the application of valuable recycled carbon fibres from cutting waste or end-of-life CFRP products via pyrolysis process. Both, flax fibres and recycled carbon fibres (rCF) have been assessed for their potential in the first test batch of WP7.2 in form of hybrid nonwoven manufactured on a small carding device in the DLR laboratory. Non-woven are semi-finished products containing randomly oriented fibres. Non-woven are a good base to mix fibres with restricted length, i.e. bio-fibres and also recycled carbon fibres (so called long fibres with a length between 6 mm to 100 mm). The mixing ratio of the fibres and also the distribution of fibres in the non-woven can be adapted according to the needs of the application. For example, the accumulation of carbon fibres in the outer layer of the non-woven should lead to an increased fire resistance and mechanical properties under flexural load compared to pure natural fibre reinforced composites. Nevertheless the use of flame retardants is of importance to meet the aviation requirements. Resin system for the first batch was a bio-based furan resin with fire properties comparable to conventional phenolic resin systems that are typically used in interior parts nowadays [7]. See chapter 4.1 for an overview of the results obtained from the first batch of tests.

As the fire properties of natural fibre reinforced composites are the most important challenge, the second batch of tests was used to assess the properties of natural fibres and rCF in combination with geopolymer (GPL) matrix prepared by the project partner VZLU. Geopolymers show exceptional fire properties and the aim was to use the positive fire properties of the geopolymer to protect the flammable natural fibres in a composite. Furthermore the application of rCF in pure and hybrid form has been assessed for potential improvement of mechanical and fire properties. Chapter 0 gives an overview of the findings in the second batch of tests.

4.1. First batch of tests

Three different variants have been tested in the first batch of non-woven reinforced composites. Two variants exhibited pure reinforcement of flax respectively recycled carbon fibre (rCF). A third sample was made of a 50/50 hybrid mix of flax and rCF. The samples were produced in the DLR laboratory on a small carding device. Difficulties resulted in the inhomogeneous distribution of fibres and a time-consuming fibre preparation. Composite plates have been produced by SLI process with a bio-based furan resin. Mechanical tests (three point bending) and fire tests (heat release, flammability, smoke & toxicity) have been carried out to assess the basic properties. The results show a considerable increase of flexural strength and stiffness by adding rCF to flax fibres in a hybrid composite. Only the pure rCF reinforced composite shows promising fire properties with the exception of high heat release. Therefore the additional use of flame retardants has to be assessed for the flax and rCF reinforced composites. The hybrid non-woven made of 50% NF and 50% rCF shows almost the same mechanical properties compared to the pure rCF reinforced sample.

Manufacturing of hybrid non-woven and composites

The hybrid non-woven were produced in a small laboratory scale device using compressed air to open (separate) fibre bundles of recycled carbon fibres and flax independently. In a second stage, the same device could be used to mix both types of fibres in the desired mixing ratio. The third step is the

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manufacturing of the non-woven in a laboratory carding device. Composites samples were manufactured from three layers of non-woven (Figure 21) with the single line infusion (SLI) method in heated hydraulic press.

Figure 21: Non-woven from 100% flax fibre (100L30) and 100% rCF (100CN25r)

<u>Fibres (1st batch):</u>

- L30: Flax (Linen) fibres cut to a length of 30mm from a sliver pre product,
- CN25r: Recycled Carbon fibres (rCF) with a maximum length of up to 25mm and fibre sizing.

<u>Resin (1st batch): Furan (bio-based thermoset):</u>

Composite fibre volume content: ~ 30%.

Specimen labelling and layering for hybrid non-woven:

- **100L30** = 100% Flax fibres L30 (L = Linen (Flax)),
- **100CN25r** = 100% recycled Carbon Fibres (rCF),
- **50L30-50CN25r** = 50% L30 and 50% CN25r.

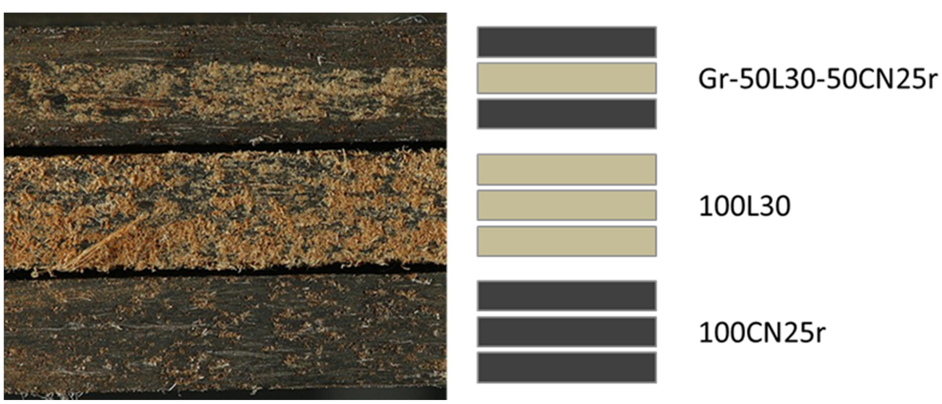

Figure 22: Photographs of the three non-woven based composites and a schematic view of their corresponding stacking sequence.

The mechanical properties have been tested by three point bending according to DIN EN ISO 14125. Figure 23 gives an overview on the obtained flexural strength and stiffness. First observation is a low flexural strength for the pure flax fibre reinforced sample. Adding 50% (volume content) recycled carbon fibres leads to a rise of almost 400% in flexural strength respectively 300% in flexural stiffness for both, 100CN25r and the hybrid variant 50L30-50CN25r. While the averaged strength and stiffness is even below the hybrid variant, the highest flexural stiffness and strength has been measured for a sample reinforced with 100% rCF. Tensile tests are expected to show a clearer distinction and should be taken into account for future investigations. An insufficient quality of the lab-scale non-woven is visible in the considerable scattering of the test results. The reason for the high scattering is an uneven distribution of fibres leading to different fibre volume content. Overall the mechanical properties measured by three point bending test are low compared to similar natural fibre reinforced samples combined with epoxy resin. Nevertheless, the results clearly show the high potential of a hybrid composite from flax and a certain amount of recycled carbon fibres.

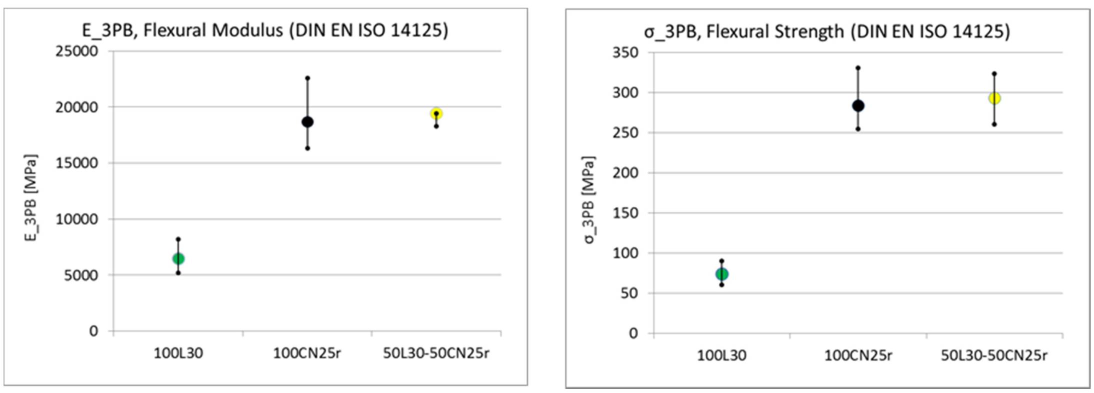

Figure 23: Flexural modulus (left) and flexural strength (right) of three non-woven variants (1. batch) with maximum and minimum values indicated.

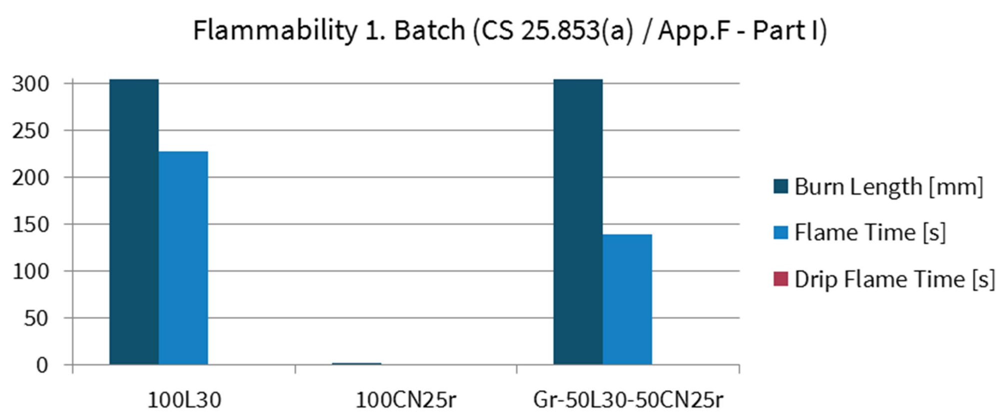

Figure 24: Flammability test results for the three non-woven specimens

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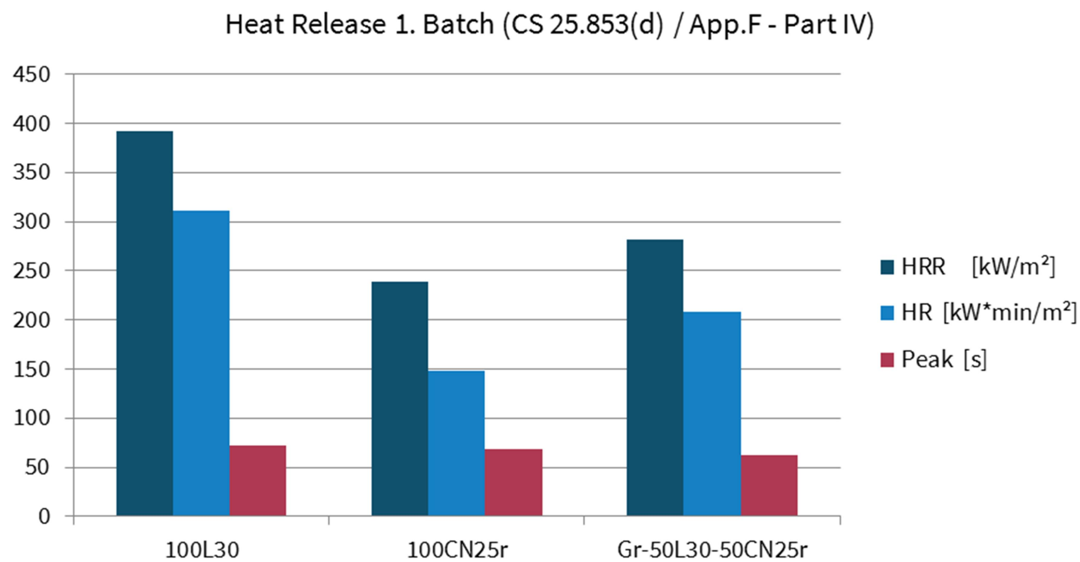

Figure 25: Flammability test results for the three non-woven specimens

Fire test results show clear trends. Only the 100% rCF reinforced composite (100CN25r) is able to fulfil the requirements for interior materials on smoke, toxicity and flammability without adding flame retardants. Flame Propagation, Smoke Density and Toxicity are well below the request threshold values [1]. Nevertheless, in order to be fully acceptable, even a 100% rCF reinforced composite with furan resin needs further fire resistance by adding flame retardants. As preceding results with glass fibre reinforcement show, the furan resin has in general the potential to substitute state of the art phenolic resins in aviation [7].

The results for the specimens reinforced with 100% flax (100L30) and hybrid of 50% flax and 50% rCF (50L30-50CN25r) reinforcement show less promising results. 100L30 has the highest heat release (HR) and heat release rate (HRR), while, as expected, the hybrid specimen has a HR between 100% flax and 100% rCF. Both samples with flax reinforcement have a burn length of 305 mm which is the maximum length restricted by the size of the test specimen. It is therefore not clearly evident if there is a difference. On the other hand the drip flame time is considerably longer for 100% flax compared to the hybrid variant (50L30-50CN25r).

4.2. Second batch of tests

Natural fibres contain mainly cellulose with smaller amounts of hemicellulose, pectin and lignin. The fire properties are a main drawback when using natural fibres as substitution for glass fibres. Therefore a combination with a matrix system that has intrinsically good fire properties could be beneficial to avoid the use of high amounts of flame retardants that typically reduce mechanical properties (matrix filler, fibre sizing) and increase the composite weight (e.g. coating).

In the first test batch, a furan resin with fire properties comparable to phenolic resin has been assessed. 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 a geopolymer matrix. Commercially available flax fabric (plain weave, 318g/m²) and a nonwoven from rCF (100g/m²) supplied by the UK based recycling company ELG have been used as reinforcement. Selected fire tests according to

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FAR for cargo compartment (F, ST, HR) and basic flexural tests will help to assess the potential advantages and challenges of these material combinations.

The panels have been manufactured by project partner VZLU in Prague with flax fabric and rCF nonwoven layup defined by DLR. 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. As the geopolymer matrix has a comparatively high density of 2,5g/cm³, it is of high importance to use fibres with a reduced density compared to glass fibres (2,5g/m³). Natural fibres (1,4-1,5g/cm³) and carbon fibres (1,8g/cm³) are therefore beneficial as reinforcements of geopolymers when compared to glass fibres. Furthermore, the typically high pore content of geopolymer composites further reduces their density to lower values than calculated.

Three test variants have been chosen: 100 % flax, 100 % rCF 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 (3PB).

Fire Properties of eco-fibres in combination with Geopolymer matrix

Flammability, Smoke & Toxicity and Heat Release test specimen have been prepared and tested at DLR site Braunschweig from the composites produced by VZLU.

Table 4: 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

Test results for Flammability and Heat Release according to FAR 25.853 are summarized in **Table 4**. A colour coding has been added to the background of the test values. 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. Nevertheless, the fluctuations 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 the GF+PF reference show a short after flame time far below the limit of 15 seconds.

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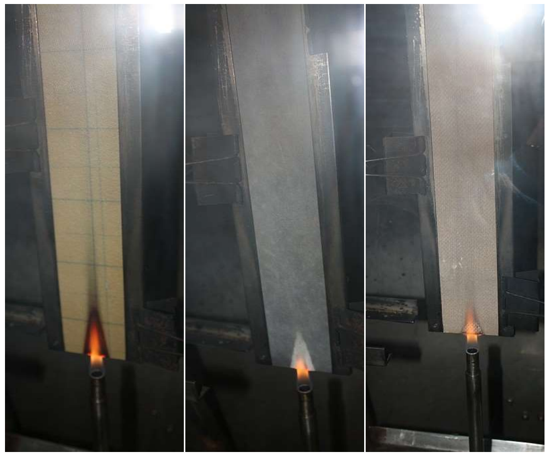

Figure 26: During the Flammability test (12s, vertical): GF+PF reference (left), rCF+GPL (middle) and flax+GPL (right).

Heat Release (HR) and Heat Release Rate (HRR) show a different picture with a clearer distinction of the different reinforcement materials (**Table 4**, **Figure 27**). Here, the advantage of geopolymer matrix compared to phenolic resin can be observed despite the use of different reinforcement materials (GF, rCF). On the other hand, the cellulosic flax fibres show their negative impact on the heat release and especially the rate of heat release. Here, 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 two 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.

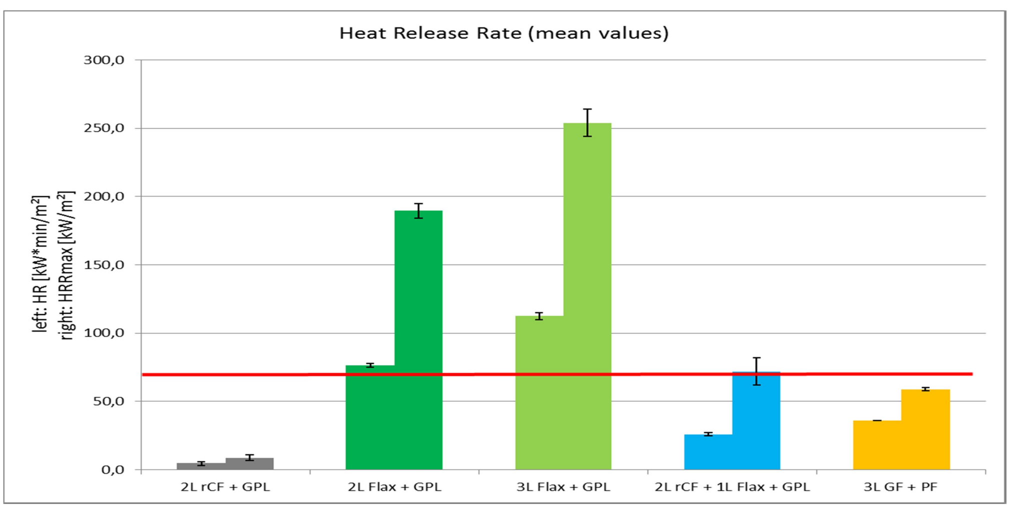

Figure 27: OSU results for Heat Release (left column for each material combination) and Heat Release Rate (right column) of the eco-fibres with GPL matrix and GF-PF reference according to FAR 25.853. The red line indicated the limit of 65 kW/m² respectively 65 kW*min/m².

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Smoke Density (S) and Toxicity (T) tests: 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 5: 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

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 three point bending test according to DIN EN ISO 14125 has been carried out by DLR. The results of the flexural tests are shown in **Figure 28**. 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. Images of the tested samples do not show any visible fracture. The flax + GPL samples show a considerable amount of plastic deformation with a strain at maximum force around 10 %. Generally, the test results are very low 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.

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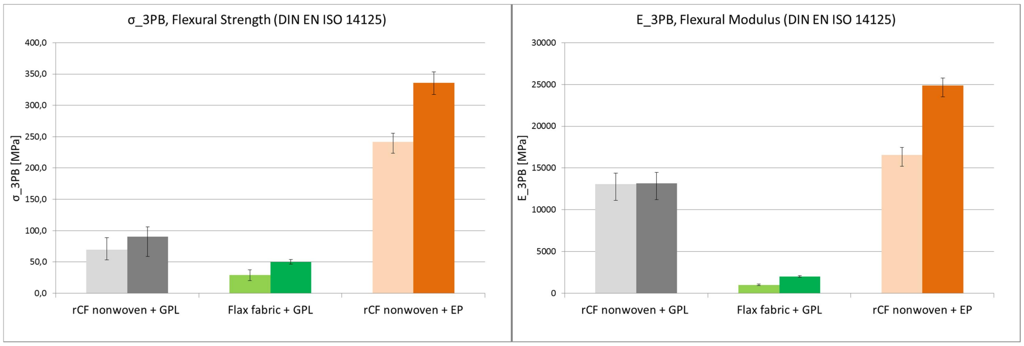

Figure 28: Flexural Strength (left) and modulus of eco-reinforcements with geopolymer (right)

5 FIBRE-METAL-LAMINATES (FML)

Manufacturing of FML is done by using prepreg technology. In advance to the layup of the laminate, the metal layers surface is pre-treated. In a first step, the metal foil is sandblasted horizontally. Hence, any contamination is removed and a rougher surface is created. After sandblasting, the metal layers will be treated with a sol-gel pre-treatment which acts as an adhesion agent. The dried metal layers are usable for the layup with the prepreg. Curing is done by within a vacuum setup and according to the standard prepreg technology. A typical vacuum lay-up for a test panel of hybrid fibre metal laminate is shown in **Figure 29**. From the cured laminate plate, specimens are produced by water jet cutting.

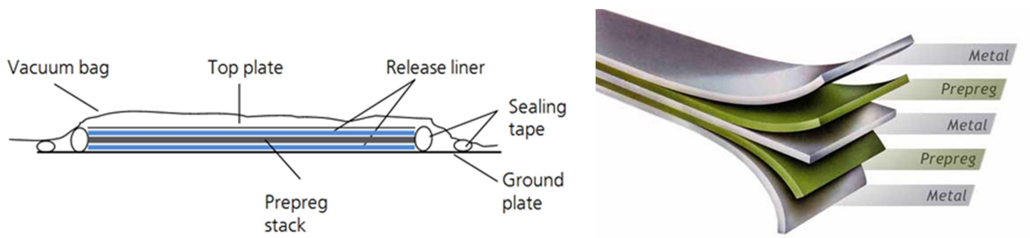

Figure 29: Vacuum setup according to prepreg technology used for FML manufacturing (left) and exemplary FML layup (right) [69]

Compared to common CFRP materials, Fibre-Metal-Laminates (FML) propose improved behavior under fire exposure. This behavior is mainly caused by the metal layers as shown in **Figure 30**. The CFRP decomposition is caused by the fire as usual. The difference to pure CFRP laminates are the Steel layers FML that do not fail themselves and act as gas barrier. Thus, less toxic gasses are released. Furthermore, the gas that is trapped between the layers insulates the rear layers from further heating. This is caused by the properties of the gas itself and by the pillow effect. The expanding but trapped gas produces pressure inside the laminate. Through that inflation process, the structure deforms like a pillow. The increased thickness acts as additional insulating factor. As a result, the rear layers will remain in a mechanical load bearing state and the structural collapse is delayed.

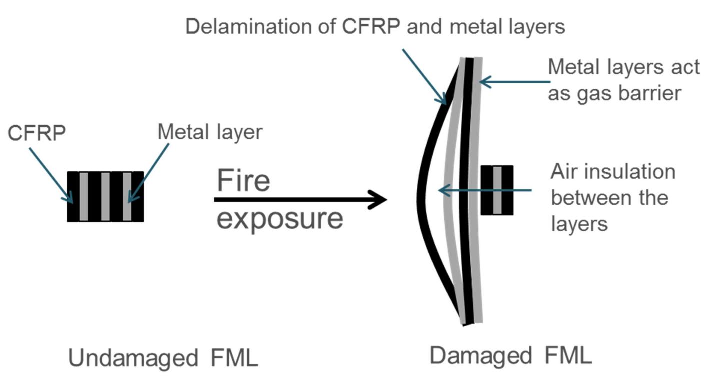

Figure 30: FML under fire exposure

To predict the mechanical behavior within a fire scenario, the temperature dependent material behavior must be known. To this, DMA measurements are conducted. The measurement comprises a continuous temperature increase with permanent mechanical loading of the specimens to measure the modulus over

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the investigated temperature range. In addition to that, measurements of properties at constant temperatures are conducted to investigate the behavior of moduli and strength. The measurements are conducted for the pure unidirectional CFRP. Furthermore, measurements are conducted for the FML to investigate the influence of the metal layers within the composite.

5.1. Fire behaviour

To investigate the behaviour of a FML within a fire scenario, subject of research were smoke toxicity and smoke density tests, as well as burn through tests with respect to varying layups. The tested laminates had a thickness of 2mm and could be divided into three test groups: varying number of metal layers (a), varying total weight of the metal layers (b) and variation of the metal layer position (c). The nomenclature was chosen by N-W-P, where N, W and P denote the number, weight and position of metal layers, respectively. The red bar named by zeros denotes the reference of pure CFRP. Figure 31, Figure 32 and Figure 33 show the results of the tests. It can be derived, that the smoke density and smoke toxicity is significantly reduced. Moreover, the number of metal layers and their position within the laminate have a high influence to the results.

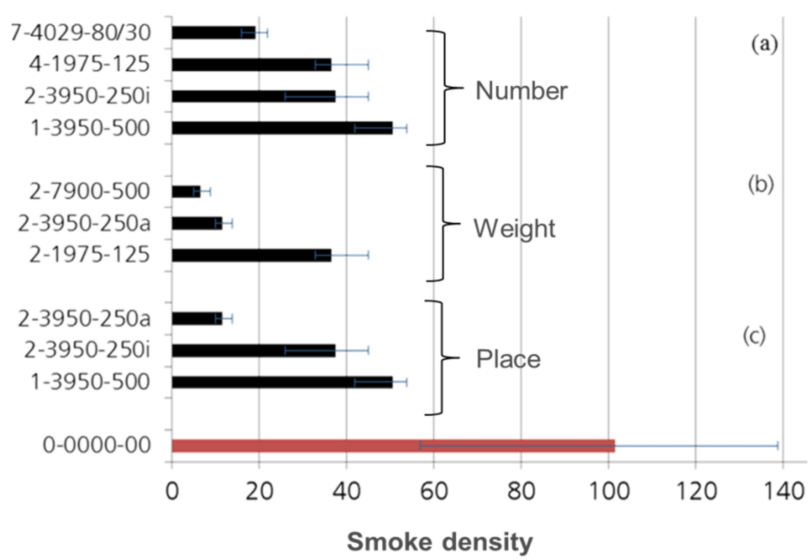

Figure 31: Smoke density test according to CS/FAR Part 25

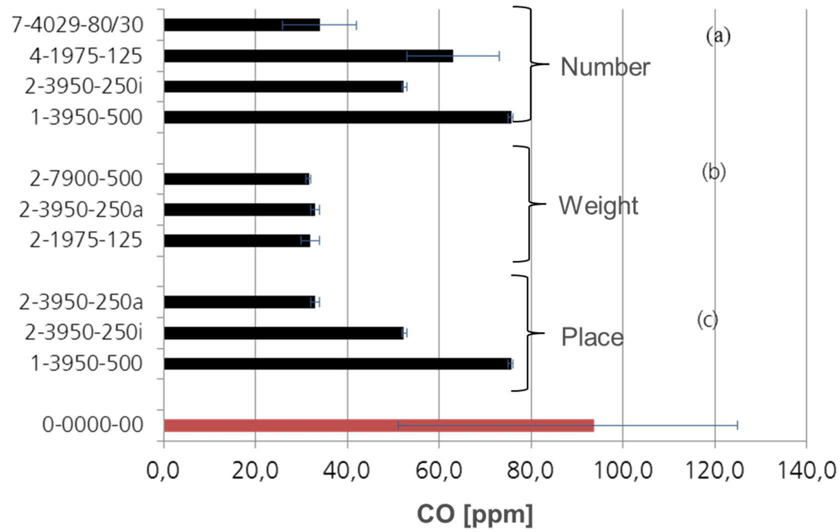

Figure 32: Results of Smoke toxicity test according to ABD 0031

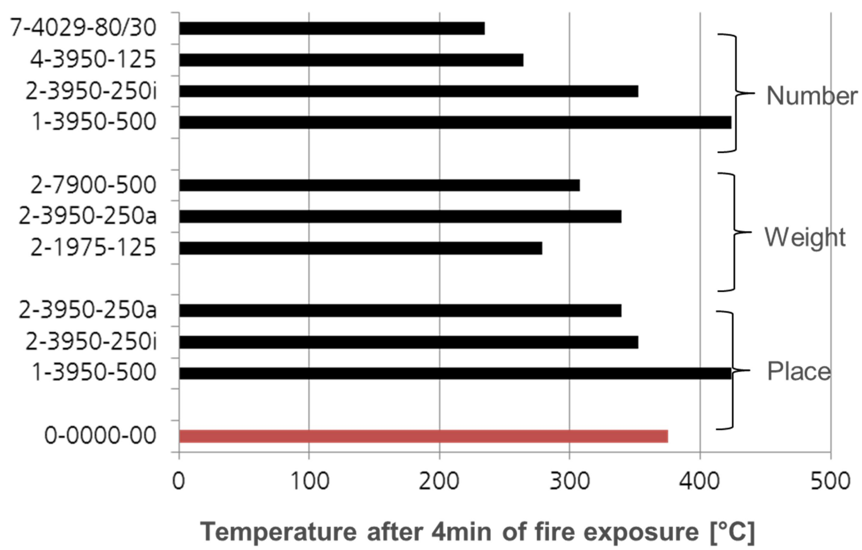

Figure 33: Burn-through test using a propane torch (Type K6 85kW)

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5.2. DMA

DMA measurements were conducted for the pure CFRP in fibre direction and transverse to the fibre. Moreover, DMA measurements were conducted for the FML. For all specimens, bending mode was used with a frequency of 1Hz. The pathway of the measured moduli over the temperature is shown in Figure 34.

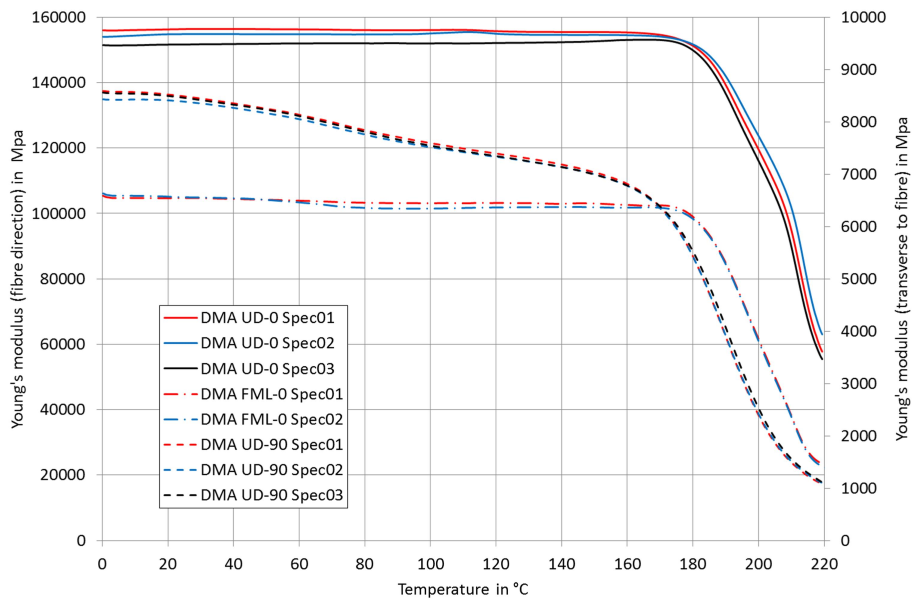

Figure 34: Results from DMA measurements: Young's modulus of CFRP and FML in fibre direction (left scale) and CFRP in transverse fibre direction (dashed lines, right scale)

5.3. Mechanical material characterization at constant temperatures

The mechanical properties were tested for the pure CFRP and the FML. It has to be mentioned that the FML has a quasi-isotropic layup containing an equal amount of UD Layers in 0° and in 90° direction. Thus, the stiffness of the FML is reduced compared to the unidirectional CFRP. The tests were conducted at 23°C (RT), 100°C, 150°C and 180°C. The tension test are conducted according to ASTM D 3039, the Compression tests are conducted using the standard of DIN EN 2850, which differentiates between specimens for modulus measurement (strain gage but no tabs) and strength measurement (tabs but no strain gage). The shear properties were determined considering ASTM-D 5379 which is using V-notched specimens. The results of the moduli are shown in Table 6. The measurements of those moduli are also conducted at 100°C, 150°C and 180°C. Unfortunately, the results are not reliable due to errors within the strain measurement which are related to adhesive properties and strain gages properties at the increased temperatures. To investigate those errors, several adhesives and strain gages were investigated. This leaded to a first guideline for testing of material properties at increased temperatures up to 200°C and higher. The guideline still has to be verified, which is currently done by testing of steel specimens and later again by cfrp specimens. The repeating of the material tests under elevated temperatures is needed. Results of the measured strengths are shown in Figures 35 and 36, and a comparison of the specimens is shown in Table 6.

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Table 6: Comparison of mechanical material properties at RT between CFRP and FML

	CFRP	FML
Tension Modulus	174120±1506MPa (fibre direction) 9110±32MPa (transverse direction)	not measured
Compression Modulus	153440±2250 MPa	102890±940 MPa
Shear Modulus	77.9±8 MPa	250.8±10 MPa

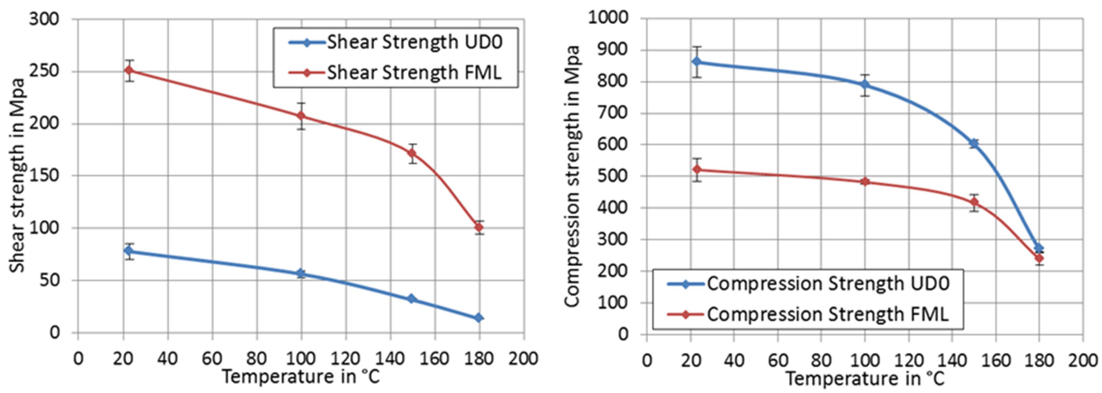

Figure 35: Shear strength and Compression strength measured for CFRP and FML depending on the temperature.

Figure 36: Compression strength representative specimens after testing – Left side: FML specimens for the varied test temperatures 23°C, 100°C, 150°C, 180°C (from left to right); Right Picture: CFRP specimen after 180°C test

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6 COMPRESSION UNDER FIRE EXPOSURE TEST (CUFEX)

Fibre-Metal-Laminates (FML) show very good behaviour regarding smoke density and burn through. However, mechanical material values have to be addressed too in case of a fire. To ensure that FMLs can withstand mechanical loads under fire exposure, a test stand was developed to load curved FML-specimens mechanically under fire exposure. It is known that the critical load case for carbon-fibre-reinforced materials is compression. The CuFEx-tests will be performed in compression with the additional fire load to resemble the most critical case.

6.1. Specimens

Compression specimens have a tendency to buckle before they reach the ultimate compression load of the material itself. The specimen geometry has to be chosen wisely to avoid early buckling. A curved specimen will support the stability of the specimen and still may be produced easily.

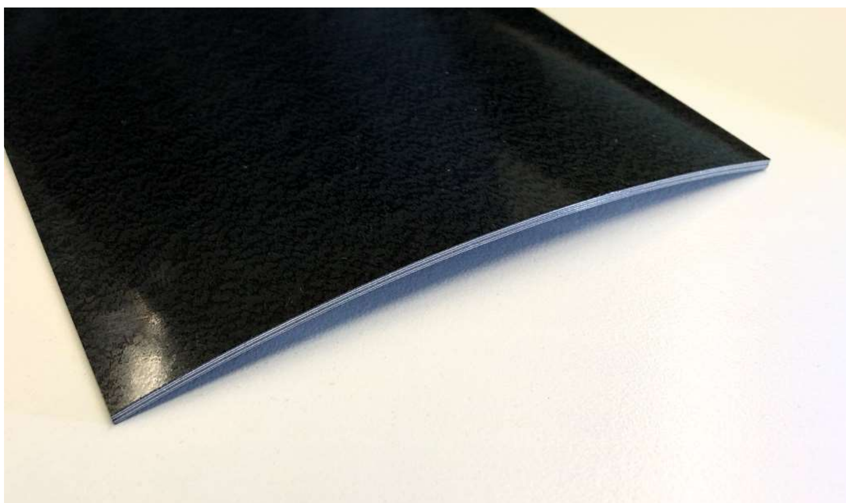

Figure 37: Curved FML-specimen for CuFex-test

The layup of the specimen is a simple combination of cross-ply layer and steel-sheets. This results in a $[[0°/90°]\ St\ [0°/90°]\ St\ [0°/90°]]_S$ layup of 2.0mm thickness. The specimens are produced by draping the layers of CF-prepreg and steel-sheets on the outside of a cylindrical mould to generate the curvature. The draped layers are cured under pressure inside a vacuum-bag in an autoclave. The tested specimens have a dimension of 200mm length and 120mm width. A length of 40mm at each side is located inside the clamping.

The specimens need a customized clamping, as there are no standardized grips for curved specimens. Also, rigid curved grips might force the specimens out of their shape and thereby introduce unwanted stresses in the material. The customized clamping has to be stiff and rigid enough to introduce the forces into the laminate and on the other hand variable enough to not introduce stresses. On top the clamping has to withstand temperatures of at least 1200°C while the specimen is flamed with a gas burner. The only feasible solution was to construct a casing into which the specimen is placed and molded by a high-temperature cement. In-plane compression loads are applied through the face of the mold. To ensure parallel load introduction, the molds are connected by installation struts. Additionally, the struts are temporarily enhanced by adjustment supports allowing precise specimen adjustment inside the first mold. The mold with the adjusted specimen is filled with concrete material. After cure of the concrete material,

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the adjustment supports are removed and the second mold is applied to the installation struts and the specimen. Again, concrete material is filled into the mold and cured. The installation struts are dismantled at the end of the mounting procedure.

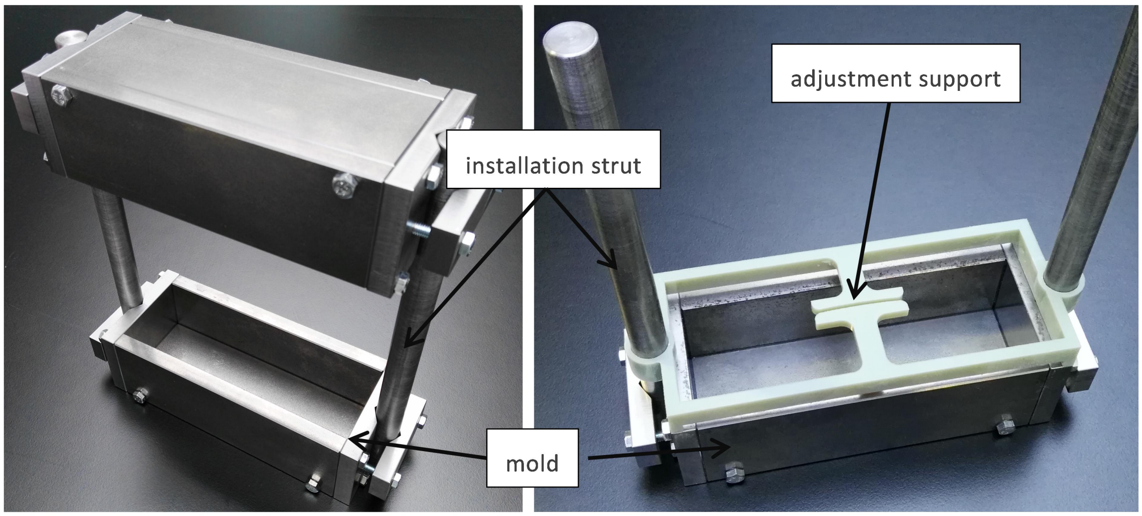

Figure 38: Specimen device; Installation struts and adjustment support are removed after moulding of the specimen

6.2. Test stand development

For the CuFex-tests a test stand had to be developed that is able to apply compressive forces onto the curved specimen while the specimen is exposed to a gas flame of 1200°C. The load must be applied along the parallel axis of the specimen without overlying momentums or forces. Hence, a guidance frame is used to guarantee a parallel guidance of the loading plates. The loading plates are guided by three high-precision struts and bearings that have “zero-play”. All components of the guidance frame are well over dimensioned to reach a maximum stiffness and a high tolerance towards transient temperature changes. Also several insulation plates and casings are installed to reduce the heat input into the test stand.

The guidance frame is installed in a standard workshop press at DLR site Trauen that has a very precise hydraulic cylinder to apply static loads to the specimen. The force is measured by a load cell that is placed between the cylinder and the guidance frame. The specimens are placed between the loading plates together with their molds.

The surface of the specimen that is exposed to the flame can be changed by using an aperture. This aperture is produced from a highly flame resistant material. The aperture is sealed against the specimen with flame-resistant wool to avoid a flow of hot gases between the aperture and the specimen. The aperture is needed to induce the “pillow-effect” in the specimen. If the specimen is tested without the aperture, the heat input is destructing the specimen before insulation gas layers can form the “pillow”.

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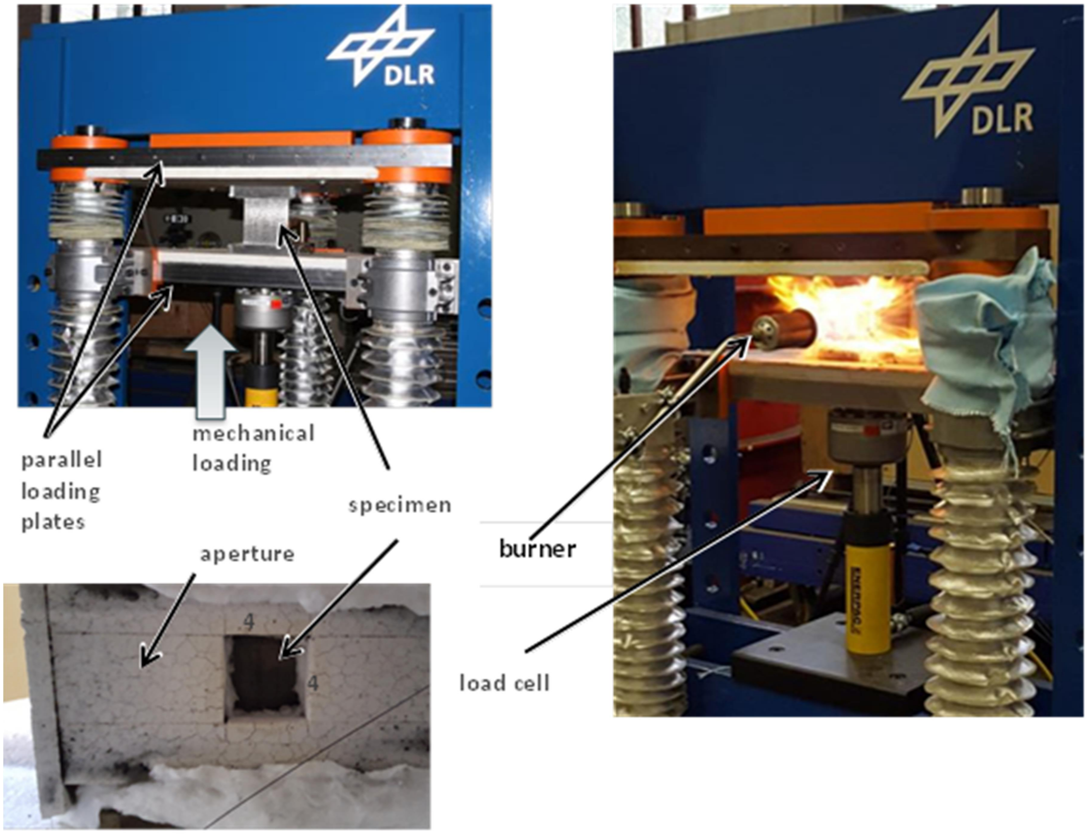

Figure 39: CuFex facility: clamped specimen (left) and while testing without aperture (right)

6.3. Results

Overall five specimens were tested in the CuFex test-stand. Four specimens them with an aperture installed and one without. All specimens withstood the mechanical preload of 50MPa without damages, but failed after they were exposed to the flame. Before each test the gas burner was calibrated and the heat flux was measured. The flame reached a temperature of at least 1200°C. The heat flux was set to approximately 200kW/m².

Specimen 01 was tested without an aperture. After the specimen was exposed to the flame, it failed after 15s. The failure occurred relatively fast, as the entire epoxy matrix of the front facing the flames decomposed and thereby withdrew the compressive stability from the specimen. The decomposition proceeded so fast, that the backside-temperature only reached approx. 100°C at the mechanical failure. The measurements were not usable to show an insulating effect of the gas layers that develop between the metal sheets of the laminate. However, this first test showed the functionality of the CuFex test-stand. Even more, it proved that no gases migrated through the laminate, even after mechanical failure.

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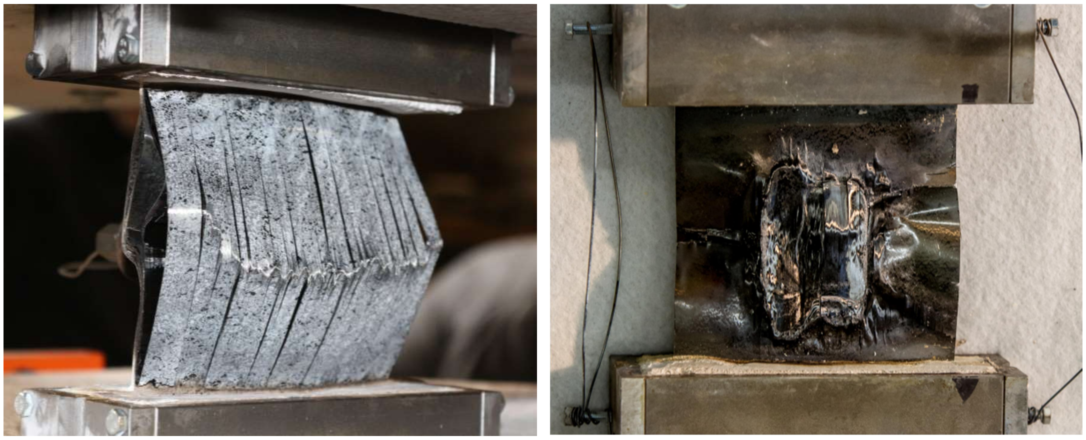

Figure 40: Comparison of specimen after test, without aperture (left) and with aperture (right)

The following specimens were tested with an aperture to reduce the area exposed to the flame. All specimens failed after more than 100s. The specimens show a "pillow-effect". This is a delamination in the material that is formed by expanding decomposition gases that push the layers outwards. The gases inside the pillow insulate the layers of the specimen and hinder the heat transition through the thickness of the laminate. This results in back-side temperature of approx. 220°C or less, even if the other side of the laminate is exposed to 1200°C. The pillow usually grows by delamination until it reaches the side of the specimen and then collapses.

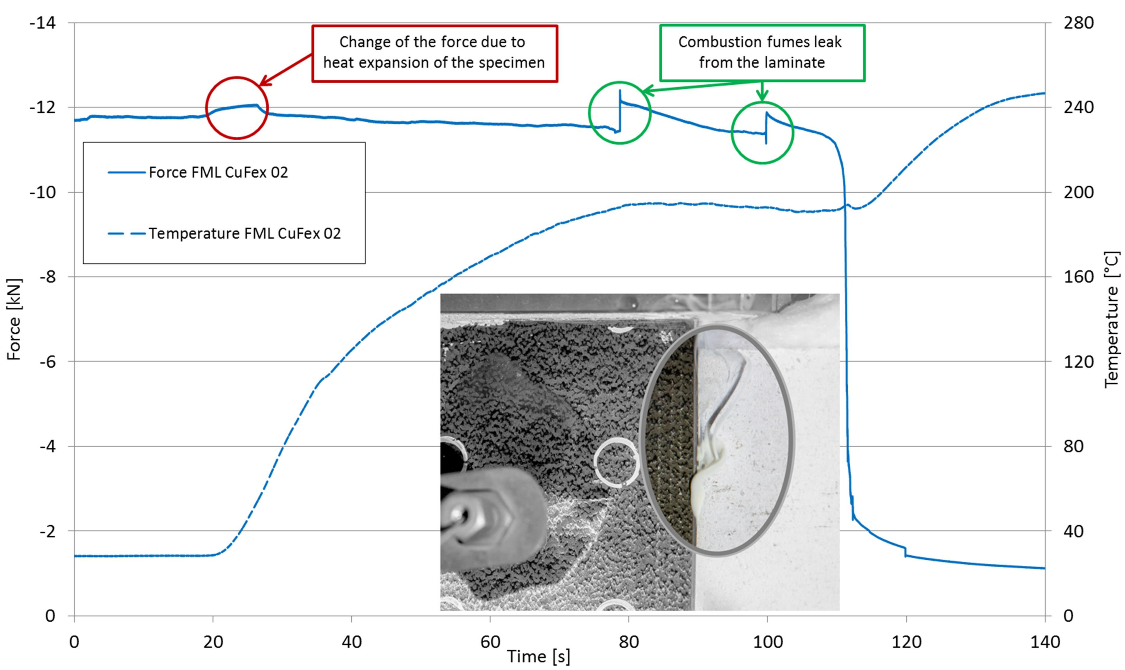

Figure 41: Back-side temperature and force plots of a CuFex-test

The curves recorded during the test of specimen 02 are shown in detail below (Figure 41). In this case the flame was put on the specimen after 20s. The heat leads to an expansion of the specimen, which subsequently pushed against the loading force. This leads to a slight rise of the load of about 200N. The

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back-side temperature starts to rise and the pillow forms on the back side of the specimen and inside the laminate. The back-side temperature rises up to approx. 200°C and remains there. 60s after the start of the flame exposure, the first pillow and 20s later the second pillow collapses. 10s later the entire specimen collapsed. After that final failure, the flame is removed. However, the back-side temperature suddenly starts to rise up to 250°C (at 120s). This may be caused by the loss of the insulating effects of the pillows.

All other test with an aperture proceeded in the same manner and an overview of the temperature-over-time plots are shown below. In this plot two different techniques of measuring the temperature are shown. The dashed lines show the measurements with an optical pyrometer. The solid lines shows the same measurements performed with a classic thermocouple that was attached to the back-side of the specimens. Two things are noticeable. First, the measurements with the pyrometer are way more dynamic than with the thermocouple, which is useful for the CuFex-tests, as the changes in the temperature may occur transiently with the collapse of the pillows. Second, the temperature measured with the pyrometer exceeds the temperature measured with the thermocouple. It could not finally be determined what the cause of this deviation is, but it seems like the thermocouple touches the specimen on a small area only. Hence it absorbs low amount heat and generates a lower reading.

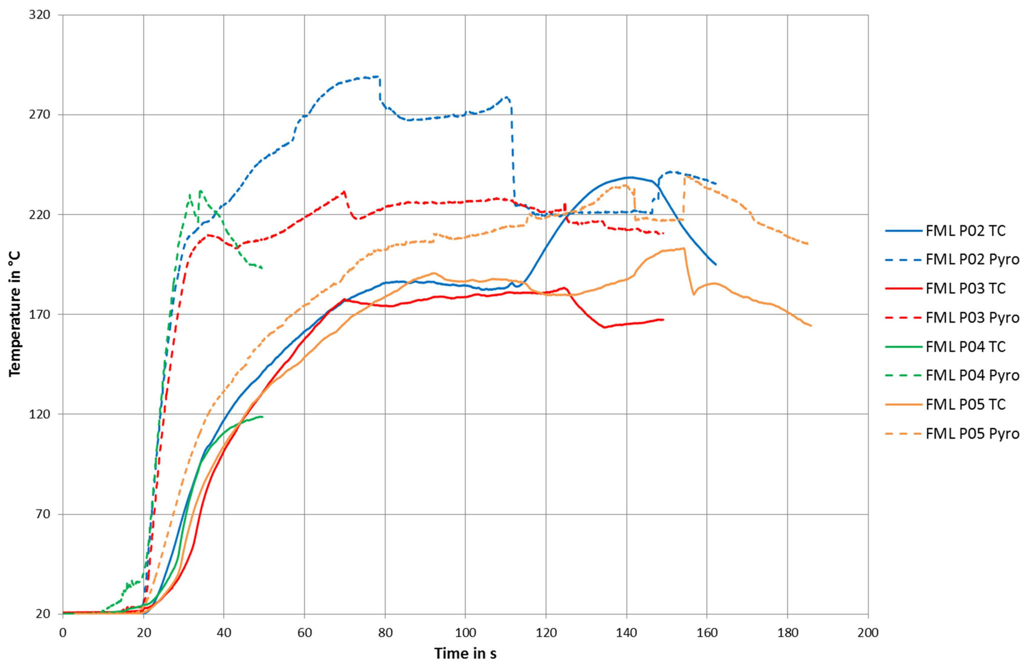

Figure 42: Back-side temperature plots of several specimen; measured with a classic thermocouple (TC) and an optical pyrometer (Pyro)

7 MODELLING AND SIMULATION

7.1. LEONARDO Modelling

7.1.1. General

The main objective of the modelling and simulation activities has been to simulate the behavior of the materials in fire condition, starting from the test results described in D7.2 , 7.5 and 7.8.

Simulation activities have been performed in order to validate the tools and improve them. In particular, the following steps have been followed:

- Verify the Tools present in house,
- Improve the tools present in house,
- Validate the tools present in house.

The simulation of the combined mechanical and fire loading supports is a main objective to improve the understanding of new material solutions. To this, temperature dependent properties measured within first and second test batch as documented within D7.5 and D7.8 should be used. Validation of the developed simulation approach is considered by comparisons to tests within the CuFex facility that are documented within D7.8.

The following two subchapters will give detailed information about the context of the tools present in Leonardo and DRL on the material behaviour in fire conditions.

- In Leonardo has been developed a tool FlamePTM with aim to simulate the flame penetration test required by CS 25,
- In DLR simulation tool has been developed to model the behaviour Fibre Metal Laminates (FML) exposed to combined mechanical and fire loading.

7.1.2. LEONARDO SIMULATION MODEL (FlamePTM)

Aim of the model is to simulate the behavior of a specimen in composite material when tested to flame penetration test.

Modelization activities have been performed in order to implementation in the model the capability to simulate the pyrolysis mechanism. In particular has been introduced in the CFD model the effect of the reaction of the gas produced by the pyrolysis mechanism with the flame, and the consequence effect on the thermo-structural behavior of the tested specimen.

The activity has been executed in the following steps:

- Experimental data examination,
- Implementation of the pyrolysis model,
- Simulation of the Thermogravimetric test analysis,
- Tool validation through numerical experimental comparison analysis..

The CFD model developed has been validated simulating the Thermogravimetric test analysis executed on G-P test specimen reported in D7.5 and D7.8.

In Figure 43 and Figure 44 comparison analysis between experimental test results and CFD model results has been shown. The model allows simulating the effect of the flame on the material characteristics due to the pyrolysis phenomena. In the model has been defined the reactions due to the pyrolysis of the solid. Gas composition has been extracted from the ABD0031 test executed by VZLU.

Analyzing the results, the little discrepancy between the two curves are due to the fact that the CFD model do not simulate in depth the preliminary material degradation due to the degradation of organic resin that starts at 300°C.

The pyrolysis model for G-P material herein described for C-GP material have been integrated in the FlamePTM.

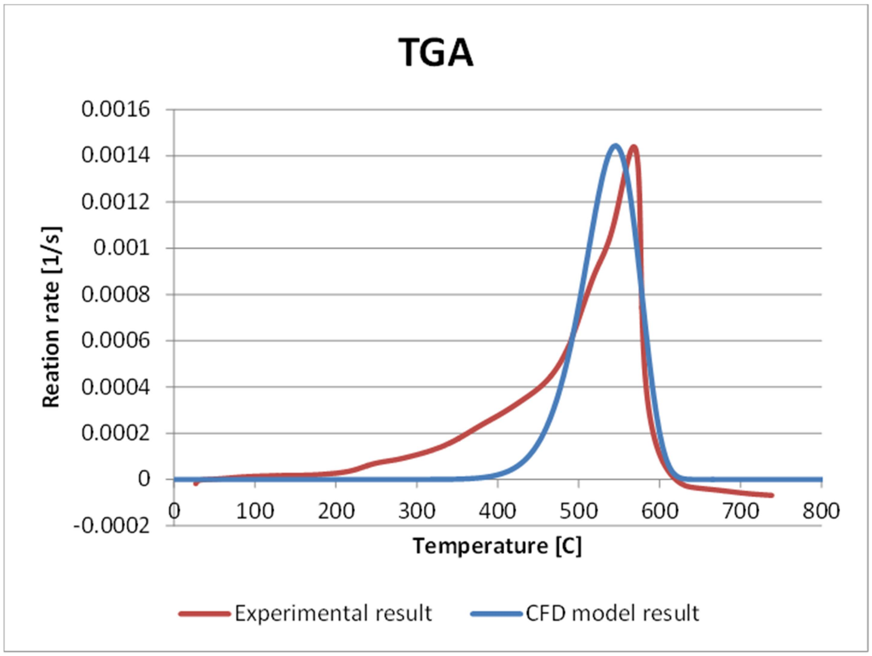

Figure 43: Comparison analysis – Reaction rate

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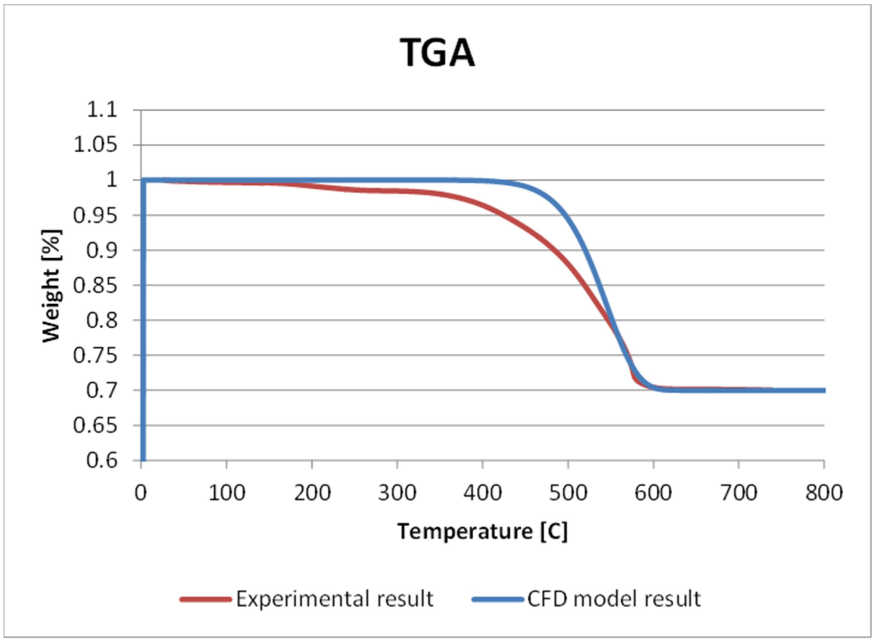

Figure 44: Comparison analysis – Loss of weight

Furthermore, FlamePTM validation activities have been carried out comparing the simulation tool results in terms of temperature field and thermo-structural behavior of test specimen with the test results.

VZLU have defined a new thermocouples installation technic in order to measure the temperature for the tool validation activities. The new technique consists in the installation of the thermocouples embedded in the test specimen (see D7.10 [3]).

In Figure 45 are shown the location of the thermocouple used during the test. In Figure 46 and 47 are shown the test results in terms of temperature in correspondence of the thermocouples. For the model validation activities the temperature measured with thermocouples 6, 7, 8 and 9 was selected. In Figure 46 and 47, the comparison analysis between the experimental test results and the model results are shown.

As shown in Figure 46 and Figure 47, FlamePTM results are more instable with respect to the test results, probably due to the different time step between the acquisition tool used during the test and the model. Furthermore, in the model, oscillation of the temperature field has been detected during the simulation of the flame generated by the burner. By the way, FlamePTM results have an acceptable correlation with the experimental results. The model thermocouples and the test rig thermocouples give the same temperature pattern.

In Figure 48, FlamePTM results compared with experimental results have been shown. The comparison analysis is only visible, the aim is to show the flame map.

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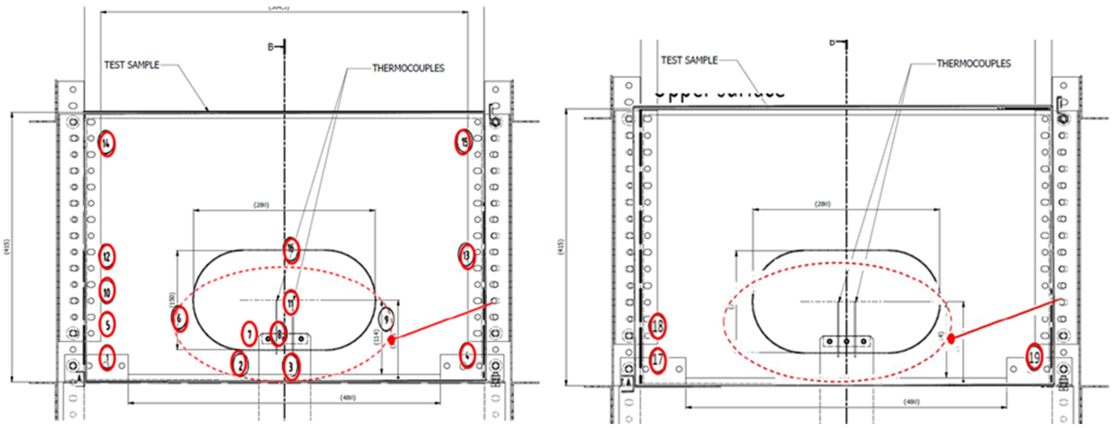

Figure 45: Thermocouples location – surface exposed to the flame on the left side

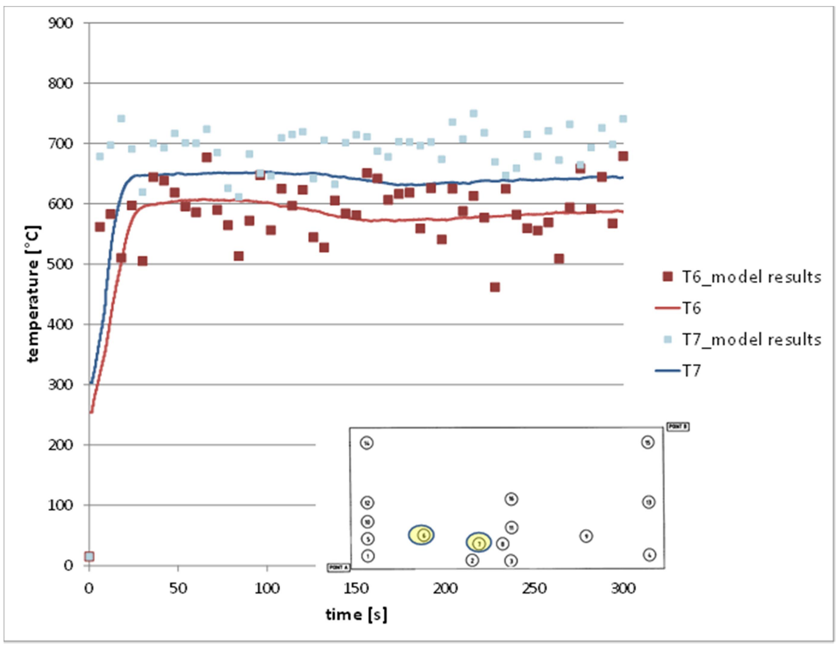

Figure 46: Comparison analysis between model results with experimental results

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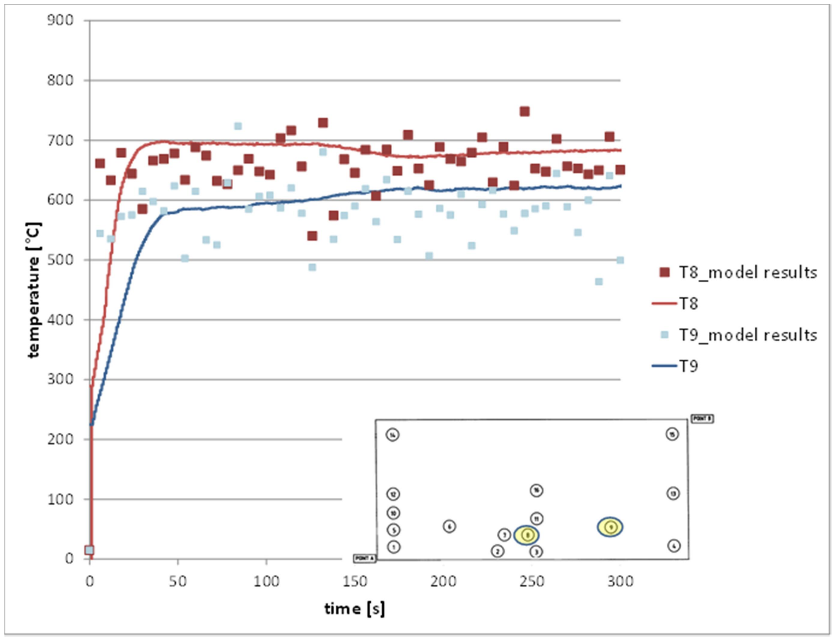

Figure 47: Comparison analysis between model results with experimental results

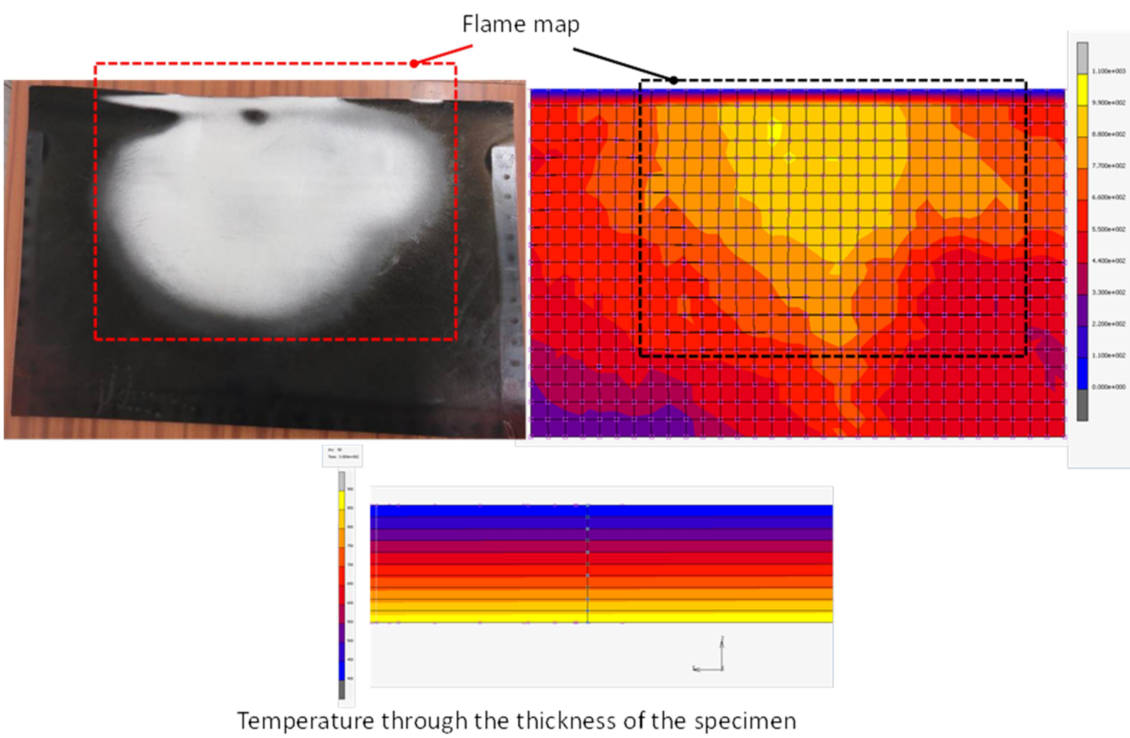

Figure 48: Temperature pattern

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7.2. DLR Modelling

7.2.1. Approach/Aim

The intention of the FE-model is to further investigate the structural behavior examined within the CuFex tests, as described within section 0. Background to that is the influence of the metal layers producing the described pillow effect (conf. section 0). However, the measurement within the composite or at any surface of the specimen is hard to achieve. Thus, the aim is to measure a few parameters with good accuracy and use them for validation of a simulation model. The simulation model will be described within the following. Its main aim is to investigate the structural behavior while combined mechanical and fire loading occur. For instance, this includes how the pillow effect develops, the propagation of cfrp material degradation and its consequences to the overall structural behavior, delamination between metal and cfrp layers and others. Finally the strategy is intended to lead to improved layups for increased fire resistance but less weight compared to the present investigated FML.

7.2.2. Modelling strategy

The modelling strategy comprises a sequential procedure to decrease numerical effort of a fully thermomechanically coupled analysis. This neglects interactions between deformations and thermal loadings. In a heat transfer analysis the temperature distribution within the specimen is calculated over the duration of the tested fire scenario. A following mechanical analysis uses the analyzed temperature distributions (obtained at each point of the specimen over the full analysis time) to determine the temperature dependent material behavior at each point and calculates the mechanical behavior of the structure. Due to mechanical degradation and thermal expansion, high deformations cause very small increment sizes. Therefore, the structural behavior over the time of fire exposure with a constant mechanical preload (test setup, conf. section **6.2**) is not calculated within the present mechanical analysis.

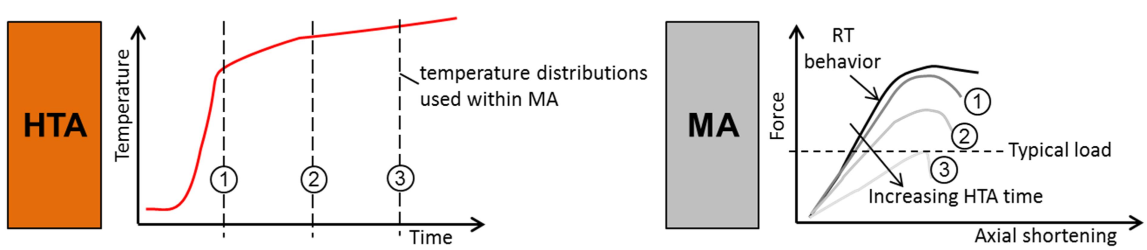

Figure 49: Modelling strategy to investigate the FML behaviour within the CuFex facility

7.2.3. Material Modelling

Within the FE-model material assignments differentiate between steel layers and cfrp layers. The steel layers temperature dependent behavior is sufficiently approximated by linear functions. The material models for cfrp are considered to describe the temperature dependent behavior within three states: the glassy state up to glass transition, the rubbery state which is past glass transition but before decomposition and the decomposed state. The glass transition temperature T_g was measured within the current studies. Measure of the decomposition temperature T_d is pending and thus it is assumed for modelling purposes. In the following, the material behavior modelled for the cfrp layers is described.

The in-plane conductivity was assumed to be constant. The conductivity in thickness direction λ_3 was measured up to T_g, and assumed to be constant within rubbery state. Past decomposition, the λ_3 is

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calculated using the ideal gas law. To this, the gas composition determined within the smoke toxicity test was used (conf. 5.1). For the decomposed state, the influence of fibres to the conductivity in thickness direction was neglected and the neat conductivity of the gas is used. To consider the thickness increase of the pillow effect and the resulting insulation, a factor was introduced. As described within section 7.2.2, the modelling strategy does not include interactions between mechanical and thermal behavior. Thus, the thickness increase through gas development (pillow effect) is not described by the present model. Thus, the introduced factor, called thickness factor $k_{T,}$ represents the thickness increase due to gas development and is added to the through thickness conductivity.

The heat capacity up to T_d was captured from DSC measurements. The decomposition energy was applied within a range of 10°C. Above that temperature, again, gas only (fibre presence is neglected) properties were assumed. The temperature dependent behavior of the heat capacity and the through thickness conductivity is visualized in Figure 50.

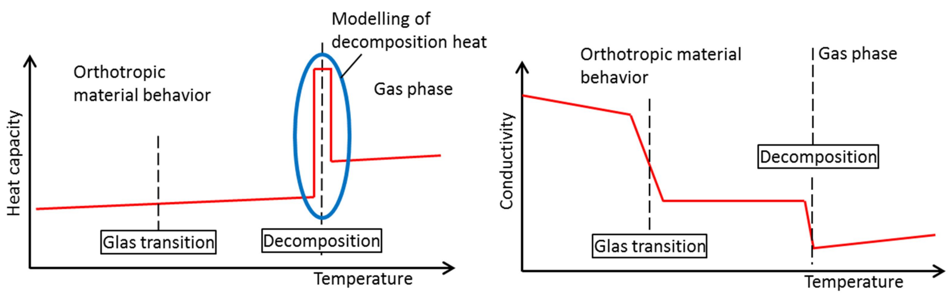

Figure 50: Schematic history of the modelled thermal material behaviour of cfrp for the heat capacity (left) and the through thickness conductivity (right)

The temperature dependent mechanical material behavior was modelled by the approach of Mahieux and Reifsnider [4]. It allows to model state transitions by the use of exponential terms. It requires the fitting through a parameter. The model was applied to the investigated cfrp materials. The studies show, that it is reasonable to introduce separate fitting parameters for each material parameter, e.g. shear or compression. Moreover the formulation of a linear factor was introduced describing the resin based reduction of a property within glassy state. By that the temperature dependency of the resin was forwarded to the laminate properties that are dominated by that behavior, e.g. shear. Summing up, each material parameter gets its separate fitting parameters and linear reduction terms and thus a properly estimated material behavior up to glass transition and past. Past glass transition temperature, the properties were assumed to reach a constant value describing the property within the rubbery state. After decomposition, the properties are set to zero. Exemplarily, the modelled temperature dependent behavior of the shear modulus is shown in Figure 51. The Figure compares the static material tests to the model proposed by Mahieux and Reifsnider [4] and a second model proposed by Gibson [4]. The models with the addition "modified" consider the resin based reduction factor as mentioned above. A more detail description to the mentioned material modelling is shown in D7.10 [3].

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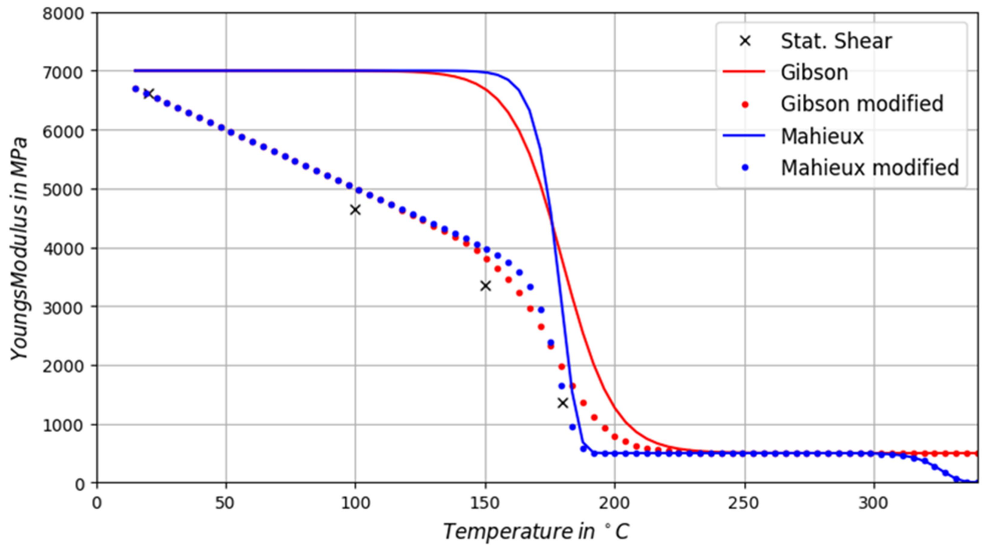

Figure 51: Comparison of material model and measurements: Shear modulus of CFRP

7.2.4. Evaluation

The comparison of the thermal model to the temperature measurements shows good agreement although several assumptions and neglections were introduced to the model. The simulation model to investigate FML behaviour within the CuFex facility uses temperature and state dependent material properties. The insulating pillow effect of the FML could be reproduced by the simulation (conf. Figure 52) and the drop of mechanical performance due to the decomposing matrix was studied (Figure 53). However, the used assumptions have to be studied further in order to more accurate prediction of the experiments Nevertheless, the basis for such deeper studies and enhancements is achieved by the present models and the conducted simulations.

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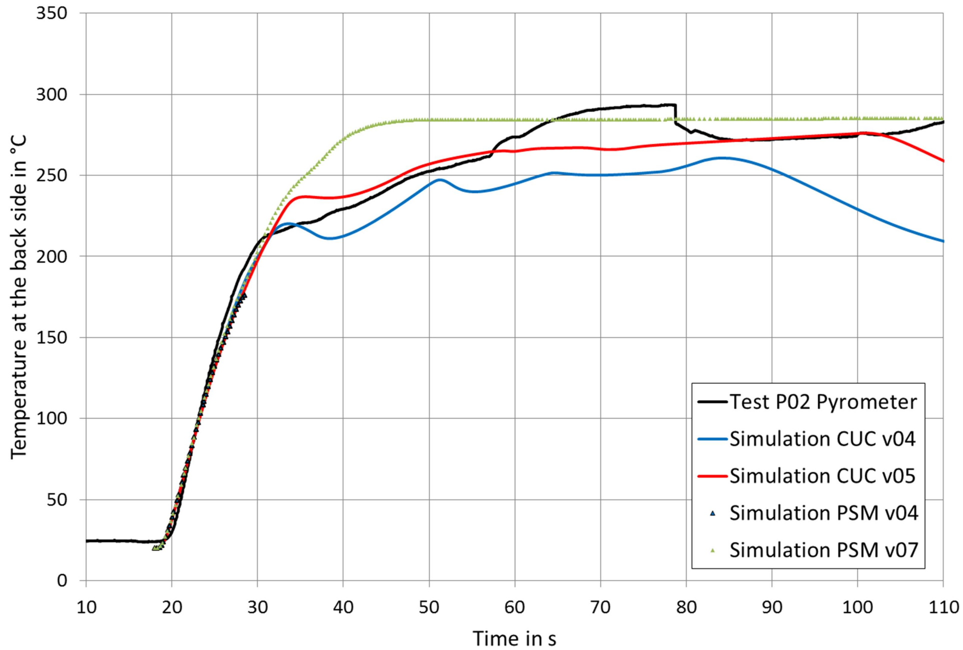

Figure 52: Comparison of CuFex test backside temperature measured by a pyrometer and the backside temperature evaluated from simulation models for the center unit cell (CUC) and the partial symmetric model (PSM)

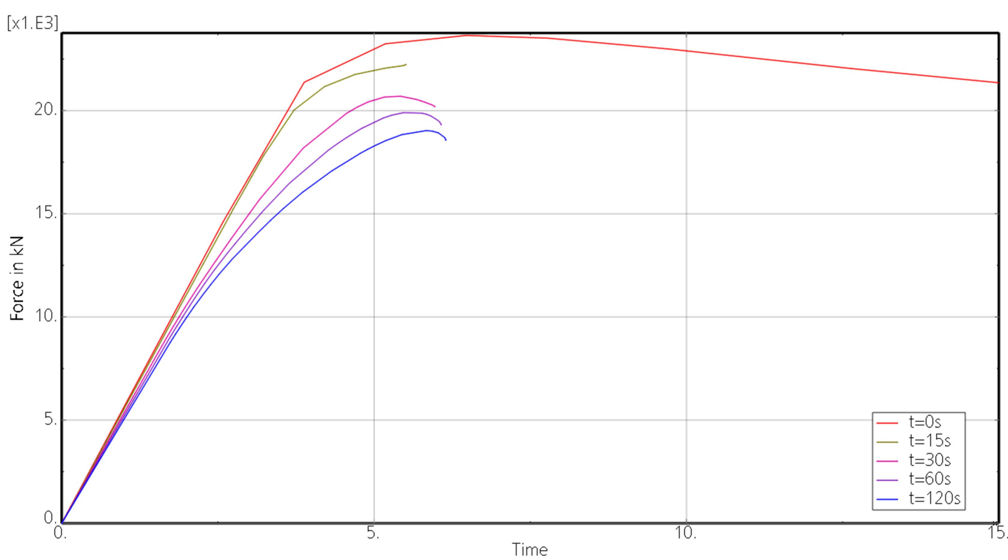

Figure 53: Comparison of load shortening curves after increasing duration of fire exposure

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8 CONCLUSIONS AND RECOMMENDATIONS

The overall objective of WP7.2 was to investigate potential of materials that may contribute to reduce the impact of fire and smoke in the cabin environment. Specific avenues that are being investigated include;

- Definition of tests to characterize material properties with respect to their fire and mechanical properties,
- Developing and characterizing of new materials and their combinations for improved fire behaviour of interior and structural materials,
- Model material degradation with respect to fire, fumes and smoke risks in the cabin environment.

Overall, two batches of tests have been planned to characterize the proposed materials and to achieve data for modelling of the fire behaviour for chosen variants. A first set of candidate new material solutions have been surveyed and some of them selected for testing in the first batch: Hybrid non-woven, Fibre metal laminates (FML), and Geopolymers (GP). The results are shown in Deliverable D7.5 [1]. Panels and specimens were manufactured for the tests (standard tests and others). Many different kinds of tests have then been performed: smoke density, toxicity, heat release, flame propagation and flame penetration tests; but also mechanical tests at various temperatures (e.g. DMA, DSC, tensile, compression and flexural tests). The first batch test results were analysed to further select and study relevant material solutions during the second batch of tests.

During the second batch of tests (D7.8 [2]), Fibre Metal Laminates and Geopolymers based material solutions have been further studied. A new approach has been considered: eco-fibres based composites (natural fibres, recycled carbon fibres and a hybrid combination of both) with Geopolymer matrix. Specimens, panels and sandwich panels have been manufactured for the tests. A specific test protocol has been developed, set up and operated (Compression under Fire Exposure, CuFex), to perform compression tests on composite panels during fire exposure. Further tests have been performed on Fibre-Metal-Laminates and Geopolymers: further fire effluents and smoke optical density tests, flame penetration test, mechanical tests (impact, drum peel, compression, CuFex). The new eco-fibre based solutions have been tested partially the same way the FML and geopolymer based solutions were in the first batch of tests to assess their characteristics and potential to be used in interior structures.

Furthermore, composition and manufacturing technique of geopolymer foam has been developed. Basic mechanical and fire resistivity tests were carried out. Geopolymer foam is a promising candidate to replace honeycombs in sandwich structures (emitting toxic products during production). Research focused on improvement of the fibre-matrix interfacial properties of geopolymer composites has been started. Wettability of fibres and fibre-matrix adhesion are factors that could improve mechanical properties of geopolymer laminates. Various methods of physical and chemical treatment of fibres were tested (plasma, acid etching, heat treatment). Precisely controlled heat treatment showed as most promising for fibres preparation.

The second batch tests were finally analysed and synthetized to compare the different solutions at material and structural levels, establish their performances and limitations, and conclude about their respective advantages and drawbacks. Chosen material combinations have also been analysed to calculate the material characteristics useful to simulate material degradation and to define the data necessary for the flame penetration model validation.

A more detailed conclusion and outlook for the different materials, the CUFEX test and modelling procedures is given in the following.

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Geopolymers (VZLU)

Carbon Fibre Reinforced Geopolymer Composite (CFRGC) was subjected to examination that included flame penetration, fire smoke toxicity (FST) and basic mechanical and impact tests. The mechanical tests were carried out with and without environmental conditioning applied (hot-wet, salt mist, working fluids).

In case of flame penetration and FST tests generally it's possible to state that CFRGC provides significantly better parameters compared to referential glass / phenol. The most expressive difference was observed at heat release tests where CFRGC provides almost 90 % lower values than referential laminates. It also features several times less production of carbon monoxide, NOx and almost zero production of HCN and other contaminants compared to the glass/phenol. In the flame penetration tests, unlike referential material, no resin ignition, smoke generation or loss of structural integrity was observed even at long-run (>15 minutes) flame expositions.

In most of examined mechanical parameters, properties of CFRGC are around to that of referential glass/phenol. Certain enhancement of CFRGC mechanical properties was achieved by improving interfacial fiber/matrix adhesion applying chemical and physical treatment of fibres. Influence of long-term environmental expositions showed similar effect to CFRGC properties as with conventional organic composites (hot water proved to be the most degrading medium).

Impact tests of CFRGC sandwich panels of the 1st batch showed noticeably worse results compared to referential ones made of standard organic materials. Application of ductile aramid fibres into CFRGC lay-up in the 2nd batch of specimens produced significantly better results. Impact resistance of hybrid carbon/aramid geopolymer has improved notably and has got near to referential material values.

Applicable CFRGC manufacturing techniques include standard methods as hand lamination, "wetpreg" or standard prepreg. Applicability of infusion production processes is limited: fabric filaments act like a filter against micro particles that the resin contains, the fabric is choked-up and vacuum penetrating process is quickly blocked.

From the point of view of material costs, CFRGC and glass/phenolic prepreg are comparable. Value approximately of 8 € per 100g of cured material (Czech Rep. 2017) can be considered as average both for CFRGC and referential prepreg. Higher price of carbon (aramid) reinforcement is compensated by low-cost geopolymer.

Ecological aspects of CFRGC material are indispensable: geopolymers feature low curing temperatures and create no by-products as phenol contaminated water or harmful vapours. Raw materials used for geopolymers preparation contains no petroleum products, poisonous contaminants, heavy metals or carcinogenic substances. Due to water solubility and non-toxicity, GP resins remainders can be drained to public canalization, as well as tools, jigs and containers can be simply washed by tap water. Geopolymer scrap – both cured and uncured - can be handled like common municipal waste.

Overall, it can be stated that geopolymer based composites are suitable, under certain conditions, to be well incorporated into aircraft structures. It is assumed that CFRGC won't be a part of load bearing airframes or other critical, particularly highly mechanically loaded components. Secondary structures as firewalls, interior linings, hot-air ducts or heat exposed elements are typical components where excellent heat and FST properties of CFRGC could be successfully utilized. The material is able to offer entirely different level of heat and FST safety at lower specific weight preserving mechanical properties and price of the state of the art solutions.

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Eco-Reinforcements (DLR, VZLU)

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. During the first batch of tests, flax and recycled carbon fibres (rCF) have been used to manufacture non-woven in the DLR laboratory. A promising bio-based resin system (furan) that exhibits comparable characteristics to state-of-the-art phenolic resin was used as matrix for the composites. Fire and mechanical tests show the potential and challenges of the different material combinations. When using 100% rCF as reinforcement, the fire and mechanical properties are very promising, while the heat release is still too high to fulfil the demanding aviation requirements. The application of cellulosic flax fibres leads to considerable challenges to fulfil the fire requirements. Therefore the additional use of fire retardants is a mandatory. Resin additives, fibre sizing and coatings need to be tested for the effectiveness and a potential negative impact on mechanical properties, environmental footprint, cabin air quality and the panel weight.

As geopolymer matrix is showing higher fire resistivity compared to classic thermoset resin systems [2], a combination of the so-called eco-fibres (flax, rCF) with geopolymer matrix has been assessed for their potential in the second test batch. Samples reinforced with rCF nonwoven show the lowest smoke density and toxicity, even lower compared to a reference composite made of glass fibres and phenolic resin. The high potential of geopolymer resin to reduce fumes and toxicity in cabin environment is therefore clearly 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 with a considerable strain. A hybrid with outer layers of rCF and an inner layer of flax shows good FST+HR results.

Generally, the weak fibre-matrix adhesion is a challenge for the use of geopolymers in combination with natural fibres and recycled carbon fibres. Cold plasma treatment to activate the fibre surface could be a solution. Furthermore, the influence of moisture must be carefully considered as 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 sensitive natural fibres 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 pore content 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 cellulosic flax fibres. It has to be explored, if a better composite quality with reduced pore content is able to improve the heat release results considerably. If not, the addition of flame retardants 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 rCF outer layers and flax inner-layers shows very promising results. Generally, for application in aviation interior linings, the highest attention should be given to the reduction of the panel weight. 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.

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Fibre-Metal-Laminates (DLR)

Fibre metal laminates (FML) were investigated to improve the present CFRP material behaviour within a fire scenario. To this, it was shown, that the metal layers acting as gas barrier significantly reduce the generation of smoke and toxic gasses. Moreover, the burn through resistance is improved allowing increased duration of mechanical performance of the structure. To further investigate this, the compression under fire exposure test was developed. Future work is needed to further improve the revealed possible effects. This includes for instance layup changes also leading to reduced weight of the laminates. Furthermore, hygroscopic effects or the effect of impacts to the behaviour within a fire scenario must be studied.

Compression under Fire Exposure (DLR)

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 mold. The concrete potting material clamps the specimen against out-of-plane deformation. In-plane compression loads are applied through the face of the mold. The tested specimens have a dimension of 200mm length, a 120mm width and a radius of 245mm. 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 structural collapse due to stability (buckling).

Five specimens were tested, four of them with an aperture and one without. The specimens with the aperture showed a “pillow-effect”, insulating layers of gases within the laminate.

The tests performed so far show the functionality of the CuFex test-stand. Now it is possible to compare different materials under fire exposure and to evaluate their fire resistance. This can be done with relatively small and simple specimens which allows to perform numerous test in a short time-span at low costs.

In the future a direct comparison of the used FML to the reference-material has to be done to quantify the pillow-effect and to get a deeper understanding of the processes of decomposition. Also the number of steel-layers should be changed to determine how many layers are needed to generate an efficient barrier for decomposition and burn-gases. Furthermore, the test could be extended by specimens with stiffeners on the back side. Such test would deliver further understanding of the insulating effect and how the mechanical performance degrades on a structural level.

Modelling and Simulation (LEONARDO, DLR)

Test results obtained during the test campaign allowed to collect all the data necessary to improve the FlamePTM characterizing in depth the test specimen material, in order to simulate and study the specimen behavior in terms of:

- Material degradation: gas produced by the pyrolysis phenomena,
- Specimen structural behavior: deformation and stress.

Structural analysis results have been not reported in the deliverable due to the negligible deformation detected with the model. It is due to the fact that the flame penetration test is a static test and the only structural effect is due to the effect of the gas produced in the specimen. It has been not considered yet in the model.

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FlamePTM with the improvement described in this deliverable assures a depth simulation of flame penetration test with the following reported important benefit for aircraft companies:

- Reduce the time and costs of specimen suppling,
- Reduced test time, the experimental activity is minimized to the confirmation of the results for the design approval,
- Reduce number of development tests and certification tests (cost reduction),
- Reduce risk associated to the development phase: the refinement is anticipated in the concept phase. (cost reduction),
- A wide spectrum of configurations and cases (optimized design).

Furthermore, a modelling strategy has been developed by DLR to investigate the structural behavior of FML with respect to simultaneous fire and mechanical loading. To decrease computational effort, the simulation was conducted by sequential heat transfer and mechanical analysis. The material models used describe the temperature dependent material behavior of thermal and mechanical properties. They are based on measurements and several assumptions. However, the comparisons of simulation and CuFex-experiment show reasonable agreement. The present simulation strategy of DLR is a good basis for future developments and enhancements such as further investigation of material properties. Moreover, within future work, the simulation is intended to be enhanced for a thermo-mechanically coupled analysis leading to the determination of improved FML layups with respect to combined fire and mechanical loading. The simulation strategy could lead to identify further improved material solutions such as optimized layups. Furthermore, this leads to a decreased effort of expensive testing.

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