





Primary structure materials – Final report on the characterisation and modelling of materials fire behaviour

J. Berthe, C. Huchette, G. Leplat (ONERA)

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

This deliverable is produced by the Project P7 "Mitigating Risks of Fire, Smoke and Fumes" of Future Sky Safety, and is a synthesis report of the work performed in Work Package 7.1 "Understanding and characterizing the fire behaviour of primary structures composite materials".

Program Manager	Michel Piers, NLR					
Operations Manager	Lennaert Speijker, NLR					
Project Manager (P7)	Eric Deletombe ONERA					
Grant Agreement No.	640597					
Document Identification	D7.12					
Status	Approved					
Version	2.0					
Classification	Public					

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



This page is intentionally left blank

ONERA Status: Approved Issue: 2.0 PAGE 2/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



Contributing partners

Company	Name
ONERA	J. Berthe, C. Huchette, G. Leplat

Document Change Log

Version	Issue Date	Remarks
1.0	24-09-2018	First formal release
2.0	28-12-2018	Second formal release (public)

Approval status

Prepared by: (name)	Company	Role	Date
Berthe, Leplat, Huchette	ONERA	Main Author	06-09-2018
Checked by: (name)	cked by: (name) Company		Date
Alex Rutten	NLR	Quality Assurance	28-12-2018
Approved by: (name)	Company	Role	Date
Eric Deletombe	ONERA	Project Manager (P7)	24-09-2018
Lennaert Speijker	NLR	Operations Manager	28-12-2018

ONERA Status: Approved Issue: 2.0 PAGE 3/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



Acronyms

Acronym	Definition
A/C	Aircraft (Commercial)
ASTM	American Society for Testing and Materials
CFRP	Carbon Fibre Reinforced Plastics
DSC	Differential Scanning Calorimetry
FSS	Future Sky Safety
JRI	Joint Research Initiative
P7	FSS Project n°7
PCRD	Program-cadre de recherche et de développement:
TGA	Thermo Gravimetric Analysis
WP	Workpackage

ONERA Status: Approved Issue: 2.0 PAGE 4/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



EXECUTIVE SUMMARY

Problem Area

An objective of the Project P7 "Mitigating the risk of fire, smoke and fumes" of Future Sky Safety (FSS) is to support increasing safety - meaning here reducing the number of casualties - with respect to fire related issues (in-flight or post-crash). First, many studies on the current flights show that about 50% of the casualties in case of aircraft accidents are linked to situations where fire is involved. Hundreds of casualties could be saved per year if fire effects on the primary structure or in the cabin environment were mitigated. Second, the development of larger, more electric and more lightweight aircraft (with an increase use of Carbon Fibre Reinforced Plastics (CFRP) composite parts in aircraft design, such as fuselage panels, engine carters, engine exhausts, ...) 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. In the FSS research program, it was decided to address the fire issue as part of Theme 4: "Building the Ultra-resilient Vehicles". It means that the research work is focused on material and structural questions, and aims at mitigating fire related safety risks when/by introducing new generation of materials in future aircraft design (incl. possible ecofriendly ones). Considering this focus, it must be noticed that very few test results are available today to the research community, because of evident costs (test facilities, destructive tests, specimens and sensors) and industry confidentiality reasons. A large part of the P7 project - to which this deliverable relates - is dedicated to develop and share experimental testing facilities and test results, with a clear partnership added value between EU Research Establishments, Academia and Industry being reached.

For new aircraft concepts, the application of CFRP is considered in the primary structure of the wing and the fuselage. Such airplane exhibits novel or unusual design features leading to a gap with the technology envisioned in the airworthiness standards dedicated to transport category airplanes. A specific concern is for safety issue pertaining to aircraft passengers with respect to crashworthiness and to fire behaviour of composite materials. Enhancing the understanding of aircraft fire performance guarantees aircraft occupants a significant safety increase to come out unharmed in case of fire incident or in crash situation. More particularly, occupant safety improvements with regard to evacuation when engine kerosene fire is developing outside will be linked to an enhancement of knowledge about the carbon epoxy materials behaviour and degradation under severe temperature conditions and fire exposure. In terms of fumes toxicity, self-estinguishibility and heat generation, the use of carbon epoxy composite materials for primary structures not only brings specific questions regarding the passengers safety, but also regarding the rescue team efficiency and safety. In terms of structures design, it is crucial to accurately understand and compare the safe, damaged (impact, crash) and decomposed (fire) materials performances, in terms of mechanical strength (load carrying) and fireproof-ness.

ONERA Status: Approved Issue: 2.0 PAGE 5/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



Description of Work

Existing testing protocols have been used, adapted, improved or new ones developed (e.g. for charred materials, for compression loadings, for tire debris impacts ...) to build up a large experimental database. The works concerned: preparation of test specimens (coupons and panels); standard tests (thermal tests at high temperatures, fire tests, ...); thermo-gravimetric tests (TGA/DSC, Fast TGA) and complementary BLADE (laser induced) tests (coupons) to operate material degradation/decomposition, measure thermal conductivity tensor and heat capacity of (virgin and) charred material. Fire exposure tests (panels); mechanical and thermo-mechanical tests (quasi-static mechanical and thermo-mechanical tests under tension and compression loadings, in the fibres, transverse and shear directions, in-plane or at the laminates interface); dynamic tests (tensile tests at various temperatures (20°C, 70°C, 120°C, and 170°C); gas gun tests for tire debris impacts on panels, followed by non-destructive testing, for latter fire exposure tests. A comprehensive analysis of all the test results has then been done: decomposition (under inert atmosphere, under oxidizing atmosphere, under dual atmosphere: inert then oxidative, under inert atmosphere at high temperature ramp), mass loss, gas phases/ species, thermal properties, thermophysical properties of charred composite laminates, damage (delamination), mechanical properties at material and structural levels (influence of the temperature, influence of the material state - virgin or charred material).

Beside the experimental works, state-of-the art models (thermal, mechanical, multi-physical) in the industry (EMBRAER) and research labs (ONERA) have been surveyed, and some of them operated and assessed based on and by comparison with the previously described test results (materials degradation, thermochemical, thermal (conductivity), thermomechanical responses, ...). The numerical methods included:

- Engineering numerical simulation approaches (EMBRAER) for the modelling of fire effects on composite structures (multidisciplinary, such as coupled CSM/CFD Computational Structural Mechanics, Computational Fluid Dynamics, etc). A simplified simulation methodology has also been proposed;
- Academic multi-physical (flame, chemical, thermal, ...) frameworks (ONERA) for the understanding of complex phenomena and the multiscale modelling of composite materials degradation and panels mechanical behaviour.

The present report is a synthesis report of the work performed within work package 7.1. It contains an overview of the experimental database produced during the project. This database is provided in the form of tables referring the specific deliverables in which the results are fully described and analysed. It also contains an overview of the modelling effort that has been done during the project. This effort was mainly dedicated to the study of "state of the art" modelling tools.

Results & Conclusions

First of all, the experimental database produced in this project is unique and provides an important number of material properties to the scientific community in order to perform and validate numerical

ONERA Status: Approved Issue: 2.0 PAGE 6/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



simulations dedicated to the fire behaviour and degradation of CFRP materials. The experimental results used to build that experimental database are described in two deliverables (D7.4 and D7.7):

- G. Leplat, A. Deudon, C. Huchette, G. Portemont, E. Deletombe, G. Roger, J. Berthe; Future Sky Safety D7.4 Primary structure materials Test Results (first batch), 2016.
- T. Batmalle, J. Berthe, P Beauchêne, V. Biasi, A. Deudon, C Huchette, P Lapeyronnie, G. Leplat, A Mavel, G. Portemont, P. Reulet, A. Palacios, R. Tejerina, J. Hodgkinson, C. Lourenço. Future Sky Safety D7.7 Primary structure materials Test results (2nd batch), 2017.

The D7.4 deliverable is mainly dedicated to the measurement of the thermal conductivity tensor and the heat capacity, the thermochemical properties and some thermo-mechanical properties of the composite material. The main conclusions were that the specific heat and thermal conductivity have been assessed as a function of temperature for the virgin state. The orthotropic properties identified from thermographic measurements have shown a significant temperature dependency. The characterisation of the thermal degradation of composite laminates needs to propose standard approach. The results concerning the influence of the heating rate on the oxidation reactions demonstrated the requirement of such procedures.

The D7.7 deliverable is mainly dedicated to the thermogravimetric analysis, the measurement of the thermal conductivity tensor and the heat capacity of the charred material, mechanical and thermomechanical testing of virgin and charred materials and decomposition of composite sample with various heating sources. Thermo Gravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) experiments had been carried out in order to identify a thermal degradation model adapted to composite materials. Heating rates of TGA measurements have also been extended to reach thermal loads of the same order of magnitude than those experienced during a fire event. Kinetic modelling assessed at low heating rates is confronted to high heating rates measurements and shows a significant local thermal nonequilibrium that requires measurements to be analysed out of the conventional thermally thin assumption. A preparation protocol to reach a fully charred (pyrolysed) state of the material has been proposed. The protocol has been successfully carried out and thermal characterizations of the charred state have been performed and are presented in this deliverable. This protocol has been also applied in order to perform the mechanical characterization of the charred material. Finally, the mechanical and thermomechanical behaviour of the virgin material have been characterized on a large temperature range in order to provide relevant data and measurements to perform coupled thermo-mechanical simulations. Laser-induced decomposition and fire exposure onto both virgin and pre-damaged coupons were tested in ONERA's facilities and extensive data were collected.

Finally, the experimental results obtained in this project have been used in order to evaluate state of the art modelling tools. This work is described in deliverable D7.9 (A. Palacios, R Tejerina, V. Biasi, G. Leplat, M.L. Rodriguez. Future Sky Safety — D7.9 Primary structures materials - Models for fire behaviour, 2017). In this deliverable, a study of the current bibliographic state of the art of thermal and thermomechanical simulations has been performed. An approach for the simulation of thermal and chemical behaviour of specific tests is proposed. Finally a simplified methodology for fire testing prediction is presented and

ONERA Status: Approved Issue: 2.0 PAGE 7/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



applied. The main conclusions were that the use of thermo-mechanical modelling in order to obtain a couple simulation of the real problem is a key factor to predict the behaviour of the composite material subjected to representative fire conditions. The analysis of simulation results can provide an estimation of the degradation growth rate of the composite materials exposed to fire as well as other boundary conditions. The analysis of simulation of the coupled problems can support the designer to take decisions related to aircraft fire wall with loads applied and to define geometries and configurations suitable from the point of view of its behaviour under generalized fire. This work has led to the following recommendations:

- i. Implement the model into suitable simulation platform software to carry out the Coupled-Simulation.
- ii. Generate a Thermo-Mechanical model by means of finite element method representative of the behaviour of materials subjected to applied loads, temperature and degradation.
- iii. Work out and analyze problems with thermal and mechanical interaction, initial thermally uncoupled followed by coupled.
- iv. Establish a systematic procedure to perform correlations of the model through results obtained in laboratory tests.

The experience related to monolithic composite fire testing when loaded through common fitting assemblies determines that the main failure occurs on the joint between load introduction device (fitting) and the composite. This joint is typically a fastened connection (bonding joint is not a non-destructively inspectable procedure and not actually accepted by EASA); the presented work has simplified the possible loading states on a fastened joint to an in-plane load state and through the plane pull out. The characterization, test and correlation with simulation of these basic configurations may be enough information to propose a simplified simulation of a complex fitting-composite configuration. A more detailed campaign could explore alternate configurations.

Applicability

The experimental database produced in this project provides an important number of material properties to the scientific community. The experimental results obtained in this project should be used in the future to feed and validate numerical simulations dedicated to the fire behaviour and degradation of CFRP materials. Moreover the recommendation concerning the thermo-mechanical modelling in order to obtain a couple simulation of the real fire case proposed in this work could be used as a starting point for numerical modelling developments.

ONERA Status: Approved Issue: 2.0 PAGE 8/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



This page is intentionally left blank

ONERA Status: Approved Issue: 2.0 PAGE 9/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



TABLE OF CONTENTS

C	Contributing partners	3
	Document Change Log	3
A	Approval status	3
Δ	Acronyms	4
Exec	cutive Summary	5
P	Problem Area	5
	Description of Work	6
R	Results & Conclusions	6
Α	Applicability	8
List	of Tables	12
List	of Figures	13
1 I	ntroduction	14
1	l.1. The Program	14
1	1.2. Project context	14
1	1.3. Research objectives	15
1	1.4. Approach	15
2 5	Synthesis of the experimental effort	17
2	2.1. Introduction	17
2	2.2. Material properties	19
2	2.2.1. Thermo-chemical properties	19
2	2.2.2. Thermo-physical properties	19
2	2.2.3. Thermo-mechanical properties	20
2	2.3. Thermo-structural behaviour	24
2	2.3.1. Thermal response – Laser-induced decomposition	24
2	2.3.2. Thermo-mechanical response – Fire behaviour	24
2	2.3.3. Standard fire test	24
2	2.3.4. Structural response	25
3 5	Synthesis of the modelling effort	27
4 (Conclusions	28
5 L	ist of deliverables produced in the WP7.1	29

ONERA Status: Approved Issue: 2.0 PAGE 10/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



ONERA Status: Approved Issue: 2.0 PAGE 11/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



LIST OF TABLES

Table 1 – Required input parameters for pyrolysis modelling	18
Table 2 - Synthesis of the thermo-chemical properties achieved in WP 7.1	21
Table 3 - Synthesis of the thermo-physical properties achieved in WP 7.1	22
TABLE 4 - SYNTHESIS OF THE THERMO-MECHANICAL PROPERTIES ACHIEVED IN WP7.1	23
TABLE 5- TEST FACILITIES AND TEST CONDITIONS FOR THE THERMO-STRUCTURAL BEHAVIOUR INVESTIGATION	26

ONERA Status: Approved Issue: 2.0 PAGE 12/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



LIST OF FIGURES

FIGURE 1 – ILLUSTRATION OF THE BLADE FACILITY: SETUP AND INSTRUMENTATION	17
FIGURE 2 – INJECT BENCH FOR CHARACTERIZING THE TOUGHNESS IN MODE II BY ENF TEST	20
FIGURE 3 – FIRE FACILITY	25

ONERA Status: Approved Issue: 2.0 PAGE 13/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



1 INTRODUCTION

1.1. The Program

FUTURE SKY SAFETY is an EU-funded transport research program 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 Program 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 Program 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. <u>SESAR</u>,). 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 Program started on the 1st of January 2015.

FUTURE SKY SAFETY contributes to the EC Work Program 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[1] [2] 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[3]. 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

ONERA Status: Approved Issue: 2.0 PAGE 14/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



 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.

1.3. Research objectives

The objective of FSS P7 work package WP7.1 "Understanding and characterising the fire behaviour of primary structure composite materials (epoxy resins, standard CFRP)" is to enhance knowledge concerning the fire behaviour and performance of CFRP primary structure composite materials, in order to better predict safety and survivability issues in case of fire incident or post-crash situation. Such predictions rely on physical models and numerical tools which need to be developed based on exhaustive materials (characterisation) and components (validation) experimental testing.

The objective of WP7.1 is to produce a comprehensive experimental database for a reference material to be shared by the European research community as a basis for material model development of the fire behaviour and degradation of CFRP materials. The T700GC/M21 material has been proposed to be used in this WP7.1 because a lot of published results already exist about its standard mechanical behaviour which the project can build on.

The objective of this deliverable is to provide a synthesis of the results obtained by WP7.1.

1.4. Approach

FSS P7 WP7.1 was split into 3 tasks:

- T7.1.1. Definition of tests, manufacturing of test coupons and panels, preparation of tests (*incl.* instrumentation), led by CEiiA (see deliverable FSS P7 D7.1),
- T7.1.2. Test and model the thermo-chemical, thermo-physical and thermo-mechanical properties
 of composite materials according to temperature, fire exposure (time), and material state (virgin
 and charred), led by ONERA,
- T7.1.3. Test and model resilience to temperature/fire effects at structural levels (incl. on damaged panels), led by Airbus D&S.

Compared to first batch of tests (see D7.4 - Primary structure materials — Test results (1st batch)), complementary tests for a second batch of T700/M21 (no confidentiality issues) specimens (coupons, panels ...) have been performed (see D7.7 - Primary structure materials — Test results (2nd batch)).

Existing testing protocols have been used, adapted, improved or new ones developed (e.g. for charred materials, for compression loadings, for tire debris impacts ...) to build up a large experimental database. The works (D7.7) concerned: preparation of test specimens (coupons and panels); standard tests (thermal tests at high temperatures, fire tests ...); thermo-gravimetric tests (TGA/DSC, Fast TGA) and complementary BLADE (laser induced) tests (coupons) to operate material degradation/decomposition,

ONERA Status: Approved Issue: 2.0 PAGE 15/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



measure thermal conductivity tensor and heat capacity of (virgin and) charred material. Fire exposure tests (panels); mechanical and thermo-mechanical tests (quasi-static mechanical and thermo-mechanical tests under tension and compression loadings, in the fibers, transverse and shear directions, in-plane or at the laminates interface); dynamic tests (tensile tests at various temperatures (20°C, 70°C, 120°C, and 170°C); gas gun tests for tire debris impacts on panels, followed by non-destructive testing, for latter fire exposure tests. A comprehensive analysis of all the test results has then been done: decomposition (under inert atmosphere, under oxidizing atmosphere, under dual atmosphere: inert then oxidative, under inert atmosphere at high temperature ramp), mass loss, gas phases/ species, thermal properties, thermosphysical properties of charred composite laminates, damage (delamination), mechanical properties at material and structural levels (influence of the temperature, influence of the material state - virgin or charred material).

Beside the experimental works, state-of-the art models (thermal, mechanical, multi-physical) in the industry (EMBRAER) and research labs (ONERA) have been surveyed, and some of them operated and assessed based on and by comparison with the previously described test results (materials degradation, thermochemical, thermal (conductivity), thermomechanical responses, etc) (D7.9). The numerical methods included:

- Engineering numerical simulation approaches (EMBRAER) for the modelling of fire effects on composite structures (multidisciplinary, such as coupled CSM/CFD Computational Structural Mechanics, Computational Fluid Dynamics, etc). A simplified simulation methodology has also been proposed,
- Academic multi-physical (flame, chemical, thermal, ...) frameworks (ONERA) for the understanding of complex phenomena and the multiscale modelling of composite materials degradation and panels mechanical behaviour.

The present FSS P7 D7.12 deliverable "Primary structure materials - Final report on the characterization and modelling of materials fire behaviour" is a synthesis report of the research works performed in WP7.1.

ONERA Status: Approved Issue: 2.0 PAGE 16/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



2 SYNTHESIS OF THE EXPERIMENTAL EFFORT

2.1. Introduction

There were two main objectives of the experimental effort of FSS project:

- 1. The first objective was to assess all material properties required to perform numerical simulations with relevant models,
- 2. The second objective was to perform extensive tests that expose the material to different types of loadings in order to help understanding the material behaviour and provide detailed database for model development and validation. Pure radiative heating was conducted using the BLADE facility (see Figure 1) and fire-induced decomposition was analysed with the multi-instrumented FIRE facility. The fire behaviour of virgin panels was investigated and compared to the fire behaviour of damaged panels which were impacted by tire debris using a gas gun facility.

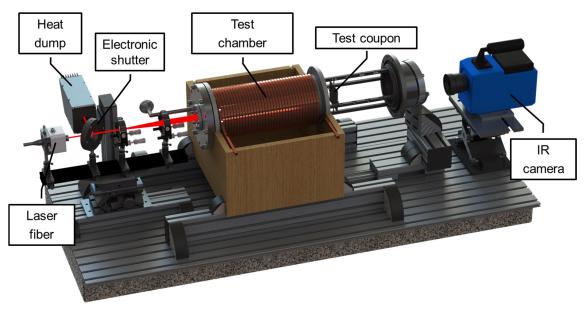


Figure 1 – Illustration of the BLADE facility: setup and instrumentation

The different input parameters required for pyrolysis modelling are listed in Table 1. The last column shows the deliverable where the results are presented.

ONERA Status: Approved Issue: 2.0 PAGE 17/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



Parameter	Method	Reaction	Species	Temperature	Decomposition state	Fibres orientation	Deliverable
Arrhenius coefficients	TGA	✓					D7.4 + D7.7
Heat of reaction	DSC	✓					D7.7
Stoichiometric coefficients	TGA	✓					D7.7
Permeability tensor	X-ray μtomography				✓	✓	Out of FSS scope
Gas properties	GC-MS	✓	✓	✓			D7.7
Density	Hydrostatic balance				✓		D7.4 D7.7
Mass fractions, volume fractions, partial densities	Homogenisation		✓		✓		D7.9
Specific heat	DSC / BLADE ONERA		✓	✓	✓		D7.4 (virgin) D7.7 (charred)
Thermal conductivity tensor	BLADE		✓	✓	✓	✓	D7.4 (virgin) D7.7 (charred)

Table 1 – Required input parameters for pyrolysis modelling

ONERA Status: Approved Issue: 2.0 PAGE 18/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



2.2. Material properties

The material studied during the project is a CFRP (Carbon Fibre Reinforced Polymer) composed of T700 carbon fibres and M21 epoxy resin. This composite material is a laminate composite material and different stacking sequences were used and analysed to assess the different properties of the material.

2.2.1. Thermo-chemical properties

The fire behaviour of composite materials is very complex because the thermal and mechanical response is combined to temperature-activated chemical decomposition. As a consequence, the first step in a properties characterization process is to assess decomposition mechanisms and kinetics as a function of temperature, heating rates and atmosphere. The conventional technique is the Thermo-Gravimetric Analysis or TGA. The experimental device used in the FSS P7 project combines the mass loss measurement with heat flux assessment in order to identify the heat of reactions simultaneously. Conventional devices have a heating rate range usually lower than $150\,K/min$. ONERA also performed TGA measurements at very high heating rates ($up\ to\ 1200\ K/min$) using a device developed for space application in order to cover the whole range of thermal loading that a material can experience when exposed to fire.

All measurements were analysed and post-processed using ADeTheC toolbox developed at ONERA in order to identify kinetic parameters and define relevant decomposition mechanisms.

The gas phase released by composite material decomposition was characterised using an isothermal pyrolyser combined by GC-MS (Gas Chromatography – Mass Spectroscopy).

A synthetic overview of the tests performed concerning thermochemical properties is detailed in Table 2.

2.2.2. Thermo-physical properties

The fire behaviour of composite materials is first driven by the anisotropic heat diffusion within the laminate due to the fibre reinforcement as a response to the heat flux generated by the fire source.

Before any decomposition occurs, optical surface properties will quantity how much heat can be absorbed by the material surface and how much heat will be lost by radiation with the surrounding atmosphere.

Specific heat and thermal conductivity components must be assessed to characterise heat diffusion within the material. Conventional techniques used for thermal conductivity can be out of their range of applicability due to the intrinsic 3D behaviour or the heterogeneous composition of such materials. As a consequence, ONERA developed a test facility dedicated to composite materials in order to assess these properties simultaneously (BLADE facility). Moreover, specific heat and thermal conductivity must be assessed as a function of temperature for all decomposition states. TGA measurements are thus used to identify the charred state of the material and to define a protocol to prepare charred coupons.

A synthetic overview of the tests performed concerning thermochemical properties is detailed in Table 3.

ONERA Status: Approved Issue: 2.0 PAGE 19/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



2.2.3. Thermo-mechanical properties

During a fire exposure the mechanical properties of composite materials evolve with respect to the temperature and to the degradation state. Most of the studies performed in WP7.1 regarding the thermomechanical behaviour were dedicated to the evolution of the mechanical properties with respect to the environmental temperature.

Regarding the mechanical properties of the laminate ply, various experimental setups have been used in order to obtain results on a large range of strain rates and temperatures: DMA testing device (1-10 Hz, 20-300°C), electromechanical or servo-hydraulic testing devices (0.5 and 100 mm/min, 20-240°C) and servo-hydraulic testing devices (5mm/min up to 2m/s, 20-170°C) equipped with climate chambers. Various UD configurations have been tested to characterise the longitudinal, transverse and shear properties of the composite material.

The properties of the interfaces between the plies have also been studied to characterise the influence of the temperature on the inter-laminar toughness. For that purpose specific ENF tests have been performed on the ONERA INJECT bench. The idea is to impose an electric current inside the material in order to increase the temperature thanks to the quite high electric resistance of carbon/epoxy laminates. With the use of this experimental facility, the interface properties have been obtained at different temperature (20°C and 80°C).

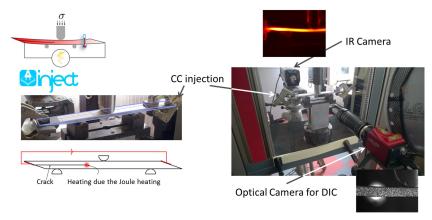


Figure 2 - INJECT bench for characterizing the toughness in mode II by ENF test

A synthetic overview of the tests performed concerning thermochemical properties is detailed in Table 4.

ONERA Status: Approved Issue: 2.0 PAGE 20/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



	Test facility	Stacking sequence	Temperature range (°C)	Heating rate (°C/min)	Atm	Deliverable
Arrhenius coefficients	TGA	UD (8ply) QI (8ply)	20-1100	1-150	N2-Air	D7.4 + D7.7
Heat of reaction	DSC	UD (8ply) QI (8ply)	20-1100	1-150	N2-Air	D7.7
Stoichiometric coefficients	TGA	UD (8ply) QI (8ply)	20-1100	1-150	N2-Air	D7.7
Gas properties	GCMS	UD (8ply) QI (8ply)			N2	D7.7

Table 2 - Synthesis of the thermo-chemical properties achieved in WP 7.1

ONERA Status: Approved Issue: 2.0 PAGE 21/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



	Test facility	Stacking sequence	Temp. range (°C)	Heating rate (°C/min)	Atm	Heat flux (kW/m2)	Exposure time (s)	Angle (°)	Deliverable
Density	Balance	UD (8ply) QI (8ply)	20						D7.4 D7.7
Specific heat	BLADE	UD (8ply) QI (8ply))	20-150		Vacuum	26	5-25		D7.4 (virgin) D7.7 (charred)
Thermal conductivity	BLADE	UD (8ply) QI (8ply)	20-150		Vacuum	26	5-25		D7.4 (virgin) D7.7 (charred)
Emissivity/ absorptivity	Spectrophotometer	UD (8ply)	150- 200		Air			0/90 Hemispheric 0/45/60/75 ≈3μm-14μm	D7.7
СТЕ	Dilatometer	UD	20-800		Air			0/90/ Out of plane	D7.7

Table 3 - Synthesis of the thermo-physical properties achieved in WP 7.1

ONERA Status: Approved Issue: 2.0 PAGE 22/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



Parameter	Method	Species	Decomposition state	Loading rate	Temperature	Fibres orientation	Deliverable
Stiffness tensor (Young Modulus, Poison ratio, Shear modulus)	Tensile machine, DMA, INJECT, climate chambers		Virgin and charred	From quasi- static to dynamic	Between 20°C and 300°C	0/90/±45 QI (8 plies)	D7.7
Fracture properties (in plane, delamination)	Tensile machine, DMA, INJECT, Microscopy, climate chambers		Virgin	Quasi-static	Between 20°C and 80°C		D7.7

Table 4 - Synthesis of the thermo-mechanical properties achieved in WP7.1

ONERA Status: Approved Issue: 2.0 PAGE 23/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



2.3. Thermo-structural behaviour

This part of FSS project was dedicated to investigate the material behaviour with an increasing level of complexity.

2.3.1. Thermal response – Laser-induced decomposition

The first step in studying composite material behaviour beyond pyrolysis activation temperature was to use the BLADE facility developed at ONERA to provide a pure radiative heating at the material surface located in a temperature and pressure-regulated test chamber. The heat source is fully controlled and characterised and the test conditions are completely regulated. The pyrolysis solver MoDeTheC developed at ONERA was validated against the experimental database provided by the BLADE facility because the thermal behaviour is isolated to be investigated separately and the fire complexity and discrepancies are voluntarily avoided. The thermal response is measured using quantitative infra-red thermography. As a consequence, the measurements are non-intrusive and do not affect the material behaviour. The BLADE facility provides a relevant framework for material model development and validation.

The effect of the heat flux magnitude and laser exposure time was investigated for unidirectional and quasi-isotropic stacking sequences.

2.3.2. Thermo-mechanical response – Fire behaviour

The second step in studying composite materials behaviour was to expose the test coupons to a laboratory gas burner while performing simultaneous measurements. The FIRE facility was developed at ONERA to investigate the interaction of the gas phase released by the composite decomposition with the flame generated by the burner. To reach this objective, the thermal response of the material unexposed surface is measured during the test while the mass loss and the unexposed surface deformation are recorded in order to correlate the temperature evolution with the decomposition reaction activation and the damage onset and growth. The gas phase release and ignition are observed using high resolution photographic images.

Virgin materials were tested in the FIRE facility as well as damaged panel that were subjected to tiredebris impact using the IMPACT gas gun facility.

2.3.3. Standard fire test

Standard fire tests have also been performed in this project by Airbus D&S. Classical test conditions have been chosen with a fire duration of 15 minutes based on specific normative conditions applicable to aeronautic structures when submitted to fire.

The temperature evolutions on the cold and hot faces have been monitored with infrared thermography. The main conclusions of these tests are: the flame did not pierce the panel during the test, there was a

ONERA Status: Approved Issue: 2.0 PAGE 24/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



high emission of fume after flame application and a small separation between fibers on flame application panel side can be observed.

2.3.4. Structural response

The objective of this specific study is to analyze the consequences of combined loads. A mechanical loading is first applied on a composite plate by means of a tire debris impact, followed by a fire loading with a laboratory gas burner. In this case, the fire behaviour of the composite plate can be modified by the possible damage generated by the tire debris impact.

For that purpose, an experimental procedure has been developed for tire debris impact onto composite panels. A gas gun has been used to perform the tire impact and the experimental design has been modified to set up displacement or strain field measurements during its impact. The modification allows to record the field measurements using the Digital Image Correlation (DIC) method during the impact test (100 m/s <impact velocity <200 m/s).

As previously mentioned, the impacted panels have been tested on the FIRE test bench (see Figure 2).



Figure 3 - FIRE facility

ONERA Status: Approved Issue: 2.0 PAGE 25/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



	Test facility	Stacking sequence	Heat Flux (kW/m2)	Exposure time (s)	Impactor speed	Instrumentation	Partner	Deliverable
Thermal response Laser-induced decomposition	BLADE	UD (8-ply) QI (8-ply)	[50-100]	[100-900]		IRT unexposed surface Mass loss	ONERA	D7.7
Thermo-mechanical response Fire behaviour	FIRE	QI (8-ply) QI (16-ply)	[50-100]	[30-180]		Stereo-DIC opposite face IRT Mass loss	ONERA	D7.7 D7.12
Standard fire test		QI	116	900		IRT	Airbus D&S	D7.7
Structural response	IMPACT	QI (16-ply)			150 m/s 171 m/s 185 m/s	Stereo-DIC opposite face Stain gauges	ONERA	D7.7

Table 5- Test facilities and test conditions for the thermo-structural behaviour investigation

ONERA Status: Approved Issue: 2.0 PAGE 26/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



3 SYNTHESIS OF THE MODELLING EFFORT

All the works performed in this work-package concerning the modelling tools have been detailed in deliverable D7.9. This deliverable contains a detailed bibliography dedicated to current state of the art of thermal and thermomechanical simulations. This bibliographic study is completed by two detailed approach proposed by ONERA and AIRBUS D&S.

ONERA's contribution to pyrolysis and fire modelling of composite materials was to perform numerical simulations using the MoDeTheC solver developed for composite applications. The solver can take into account:

- anisotropic heat diffusion within heterogeneous materials such as composite materials,
- energetic contribution of (endothermal or exothermal) decomposition reactions,
- gas phase formation and transport within an anisotropic porous medium,
- anisotropic thermo-physical properties evolution as a function of temperature and decomposition state using advanced homogenisation methods to define bridging function between virgin and charred states during decomposition.

AIRBUS D&S's contribution relies on a simplified methodology for fire testing prediction. This methodology is applicable to the prediction of standard fire tests on composite loaded panels. This methodology relies on:

- Simplified thermal boundary conditions:
 - o Flame model, based on the repetitivity of thermal hot face map on a standard fire test,
 - o Convection model based on constant heat transfer coefficient dependent on the cold face flow speed.
- A thermal characterization on an intact panel and a degraded panel,
- Simplified assumption of a direct relation between thermal and mechanical degradation,
- Simplified failure criteria based in cold face temperature map evolution through test,
- Confirmation, by dedicated and especially monitored test, of the adequacy of these assumptions for a typical composite configuration in a typical load range,
- Thermal (only) transient problem resolution to establish the cold face temperature map evolution,
- · Determination of the failure instant based in the failure criteria defined,
- Thermomechanical problem resolution, with the same assumptions, to evaluate impact of secondary effects that may impact on the problem (large deformations with impact on thermal contact between elements).

ONERA Status: Approved Issue: 2.0 PAGE 27/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



4 CONCLUSIONS

The present synthesis report gives an overview of the research effort done in the work package 7.1 of the Project P7 "Mitigating the risk of fire, smoke and fumes" of Future Sky Safety (FSS).

The first main objective of WP7.1 was to produce a comprehensive experimental database for a reference material to be shared by the European research community as a basis for material model development of the fire behaviour and degradation of CFRP materials. The present synthesis report can be used as a reference document to share the results within the European research community. All the experimental results obtained in the work-package are summarised in the present report tables. For each property studied, a clear reference is made to the different documents in which the methodology, the experimental results and the analysis are described.

The second main objective of WP7.1 was to confront experimental results to state-of-the-art models and simulation tools. As mentioned in this report, a specific deliverable (D7.9) was produced. This deliverable contains a detailed bibliography dedicated to current state of the art of thermal and thermomechanical simulations. This bibliographic study is completed by two detailed approaches proposed by ONERA and AIRBUS D&S.

Five main objectives have been defined for the Future Sky Safety Project P7:

- O1: Improving knowledge concerning OMC materials and structures behaviours vs fire,
- O2: Assessing mechanical properties of heating/burning/degraded materials/panels,
- O3: Evaluating the heat/fire consequences (incl. toxicity, smoke), proposing solutions to mitigate them,
- O4: Sharing database for future modelling purposes (expensive tests),
- O5: Establishing/giving design recommendations.

This report illustrates that the research effort performed in this work-package have contribute the achievement of at least three of these objectives. First, the experimental results have increase the knowledge concerning OMC materials and structures behaviours vs fire. Secondly, the results obtained in this work-package allow assessing the mechanical properties of heating/burning/degraded materials and/or panels. Finally, as previously mentioned, this synthesis report is a sum up of the results that can be used to share the experimental database for future modelling purposes.

ONERA Status: Approved Issue: 2.0 PAGE 28/29

Reference ID: FSS_P7_ONERA_D7.12

Classification: Public



5 LIST OF DELIVERABLES PRODUCED IN THE WP7.1

- D7.1 M. Oliveira, J. Berthe, G. Leplat, C. Huchette, A. Sanz Rodrigo, R. Reis. Future Sky Safety D7.1 Plan of Experiments Primary Structures Materials Final Requirements, Selection and Specification of Materials and Tests, 2015.
- D7.4 G. Leplat, A. Deudon, C. Huchette, G. Portemont, E. Deletombe, G. Roger, J. Berthe. Future Sky Safety D7.4 Primary structure materials Test Results (first batch), 2016.
- D7.7 T. Batmalle, J. Berthe, P Beauchêne, V. Biasi, A. Deudon, C Huchette, P Lapeyronnie, G. Leplat, A Mavel, G. Portemont, P. Reulet, A. Palacios, R. Tejerina, J. Hodgkinson, C. Lourenço. Future Sky Safety D7.7 Primary structure materials Test results (2nd batch), 2017.
- D7.9 A. Palacios, R Tejerina, V. Biasi, G. Leplat, M.L. Rodriguez. Future Sky Safety D7.9 Primary structures materials Models for fire behaviour, 2017.
- D7.12 J. Berthe, C. Huchette, G. Leplat. Future Sky Safety D7.12 Primary structure materials Final report on the characterisation and modelling of materials fire behaviour, 2018.

ONERA Status: Approved Issue: 2.0 PAGE 29/29