Characterization and modeling of thermo-mechanical behavior of aeronautical CFRP materials exposed to fire.

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1. Abstract

This paper describes first, small scale tests on carbon epoxy laminates exposed to a flame, simulating post-crash fire or in flight fire. Secondly, a thermal model has been developed to evaluate temperature profile and pyrolysis zone in the bulk of carbon epoxy laminates and compared to experimental results.

Safety aboard aircraft is one of the main concerns of aircraft manufacturers and airline companies. For 20 years, the fire threat in aeronautics has been decreasing, but, the intensive use of polymer composites in new generation aircraft (A350 or B-787 families), for weight reduction and energy saving purposes, causes the fire scenarios to evolve in terms of threats. The global aim of AircraftFire (AcF) was to contribute to the evaluation of the evolution of the fire threat linked to the substitution of aluminum alloy by composite materials in major aircraft structures and its consequences on the survivability of occupants in the new generation of aircrafts.

Mainly CRFP monolithic composite with thermoplastic or thermoset matrix for aeronautical structures provided by Airbus were investigated, but this paper focused only on results and modeling related to carbon fibers/epoxy matrix laminate composites. For this purpose a thermo-mechanical bench has been designed and built by Institut Pprime, coupling a bending machine and a propane/propylene burner or cone calorimeter. The composite materials were first bended at room temperature and exposed to a constant heat flux until the failure. Thermo-mechanical behavior of the composite materials was investigated for four calibrated heat fluxes representing different fire scenarios: 75, 106, 155 and 200kW/m². For each test, temperature was measured at three different locations on the back side of the sample. Some tests were performed in unload condition for two different times, then the samples were scanned by X ray tomography in order to evaluate the pyrolysis zone induced by thermal degradation. TGA experiments were performed to describe resin pyrolysis, DSC tests to measure the temperature of glass transition and heat capacity as DMA tests describe the temperature modulus evolution and the transition from elastic to visco-plastic behavior.

In order to model the experiment, extensive work was performed to determine by inverse method and optimize thermal properties of composite material including pyrolysis effect. Based on back side specimen temperature measurement with different Heat Flux (75kW, 106 kW and 155kW), on specimens thicknesses of 2mm and 4mm, at mid span and 80 mm far, combination of all these temperature evolution have been used to determine and optimize heat capacity and thermal

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conductivity by inverse problem. An algorithm has been developed based on the optimization software Dakota.

2. Introduction

Safety aboard aircraft is one of the main concerns of aircraft manufacturers and airline companies. For 20 years, the fire threat in aeronautics has been decreasing, but, the intensive use of polymer composites in new generation aircraft (A350 or B-787 families), for weight reduction and energy saving purposes, causes the fire scenarios to evolve in terms of threats. The global aim of AcF program was to contribute to the evaluation of the evolution of the fire threat linked to the substitution of aluminium alloy by composite materials in major aircraft structures and its consequences on the survivability of occupants in the new generation of aircrafts. The consortium supported this large project was composed of 13 partners, 9 universities and 3 industries. AcF program was divided in 4 parts mainly oriented in fire properties and modeling, covering Fire Threat Analysis, Aircraft Fire Prevention, Aircraft Fire Protection and Aircraft Simulation Tools. One work package of this program was considering the effect of fire on mechanical properties of composite material in case in Post-crash fire condition or in flight fire.

3. Materials and experiments

3.1. Materials

Structural composite materials provided by Airbus were tested combining different type and layup of carbon fibers, type of matrix in two different thicknesses (around 2 and 4mm), with fiber volume content in the range 55% to 58% (cf. table 1). Due to confidentiality of materials used in the project, a consortium agreement has been signed firmed between all the partners, so commercial names have been replaced by AcFx.

Reference	Fiber	Matrix	Thickness (mm)
AcF1	Carbon UD	Epoxy	2,1 or 4,19
AcF2	Carbon Woven	Epoxy	2 or 4,02

Table 1 : AcF materials tested

3.2. Experimental device

A mechanical bench has been designed and built by Institut Pprime specifically for AcF program [1]. The main idea to design this bench was to allow doing tests with the sample face in front of the burner, either in tension or in compression, keeping all the other experimental parameters constant. The equipment allows 3 or 4 points bending test on 50mm width and 300mm length specimen and is coupled with a burner. The burner has a wide range of heat flux from 75 up to 200kW/m² over a diameter of 3.5cm, and a maximum temperature gas exhaust of 1200 ° C (Photo 1). Previously, the heat flux was measured at the nozzle distance corresponding to the bended sample position (52mm). For each test, the burner was ignited first, the exhaust temperature controlled until the stabilization. Test procedure developed is the following, the specimen is first bended at room temperature to a fixed deflection, then when the heat flux is reached and stabilized, the bench slide in front of the burner, starting the experiment. As soon as the specimen is broken, the bench is removed from the burner. A thermocouple of 0,5mm diameter is fixed in front of the specimen in order to determine accurately the initial time t0. Otherwise, three thermocouples of 0,5mm diameter measure the temperature on the rear side of the specimen at three locations, mid span (0mm), 10 and 80mm from the mid span.

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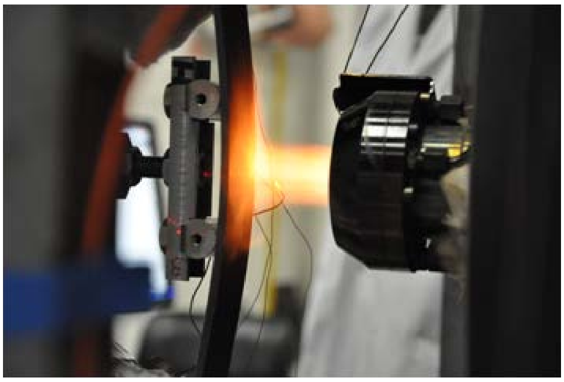

Photo 1 : Experimental device – Mechanical test of carbon epoxy laminate exposed to a methane burner flame

3.3. TGA and DSC experiments

Modeling the thermo-mechanical behavior of composite material required the knowledge of multiple phenomena as decomposition of the matrix, evolution of the mechanical and thermal properties with the temperature. For this purpose, TGA experiments were performed in Nitrogen atmosphere at three heating rates of 5°C/min, 10°C/min and 20°C/min from the ambient temperature (20°C) to 1000°C. Experimental curves show that ACF1 and ACF2 degrade similarly in a one single-step. DSC experiments were performed from -20°C to 250°C, in order to measure the evolution of the specific heat (C_p) and the glass transition temperature (T_g) of the matrix. DMA tests were performed from 20°C to 250°C in order to describe the temperature modulus evolution and transition from elastic to visco-plastic behavior of the matrix, witch correspond to a sudden drop of stiffness.

4. Experimental Results

4.1. Bending test under fire

As the stacking sequence of each composite material is different, a calculation of the stress induced by the bending displacement must be done in order to further compare the behavior of each one. For this purpose, FEM models of AcF1 and AcF2 have been realized to calculate the stress field across the specimen. The calculated resultant load on the specimen was compared to experimental bending tests performed at room temperature. In order to give an idea, for AcF1 and AcF2 (4mm thickness, 15 mm bending deflection), the maximum stress calculated in the length direction, across the thickness reach about 276,80MPa, but is not located in the same plane due to stacking sequence differences. According to the ASTM standard D 790-07 [2], in case of span to thickness ratio greater than 16 to 1, the stress in the outer surface of the specimen at midpoint (S) can be reasonably approximated with the equation (eq.1):

$$S = 3 * P * L/2 * b * h\,[(1 + 6(D/L)^2 - (4(d/L)(D/L)]\ \text{(eq. 1)}$$

Where P: load (N); L: support span (mm); b: specimen width (mm); h: specimen thickness (mm) and D: deflection of the centerline at the middle of the support span (mm)

For AcF2, stresses in the outer surface at mid-span at room temperature calculated by equation (eq.1) are presented in the Table 2, showing a discrepancy between FEM models and the analytic result of about 7% for AcF2, 4mm thickness, 15 mm deflection.

AcF2	Mid span deflection 15 mm	Mid span deflection 30 mm
Thickness 2mm	149,25MPa	275,71MPa
Thickness 4mm	296,04MPa	

Table 2 :Stress in the outer face at mid-span for AcF2 for different bending deflection

Fire tests under bending solicitation were performed on AcF1 and AcF2 with extended experimental conditions covering:

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- Four heat fluxes : 75kW/m², 106kW/m², 155kW/m² and 200kW/m²,
- Two different thicknesses : 2 and 4 mm
- Two bending displacements: 15 and 30 mm,
- Face exposed to the burner in tension or in compression.

Over 50 experiments have been performed during this program with the burner in order to get general outline of the composite materials mechanical behavior submitted to flame impact. For all these tests, the distance between the burner and the sample was kept at 52mm. During the test, the bending deflection remains constant as the load evolve, until specimen failure. As the breakage of the sample is not obvious to determine, we consider the failure time as the time calculated by linear regression of load drop occurring during the test. Figure 1 presents the failure time for AcF1 and AcF2 for two thicknesses (2 and 4mm), two deflections (15 and 30mm) inducing tension stress for the face exposed to the flame. It is important to note that during the tests, no specimen caught fire before failure.

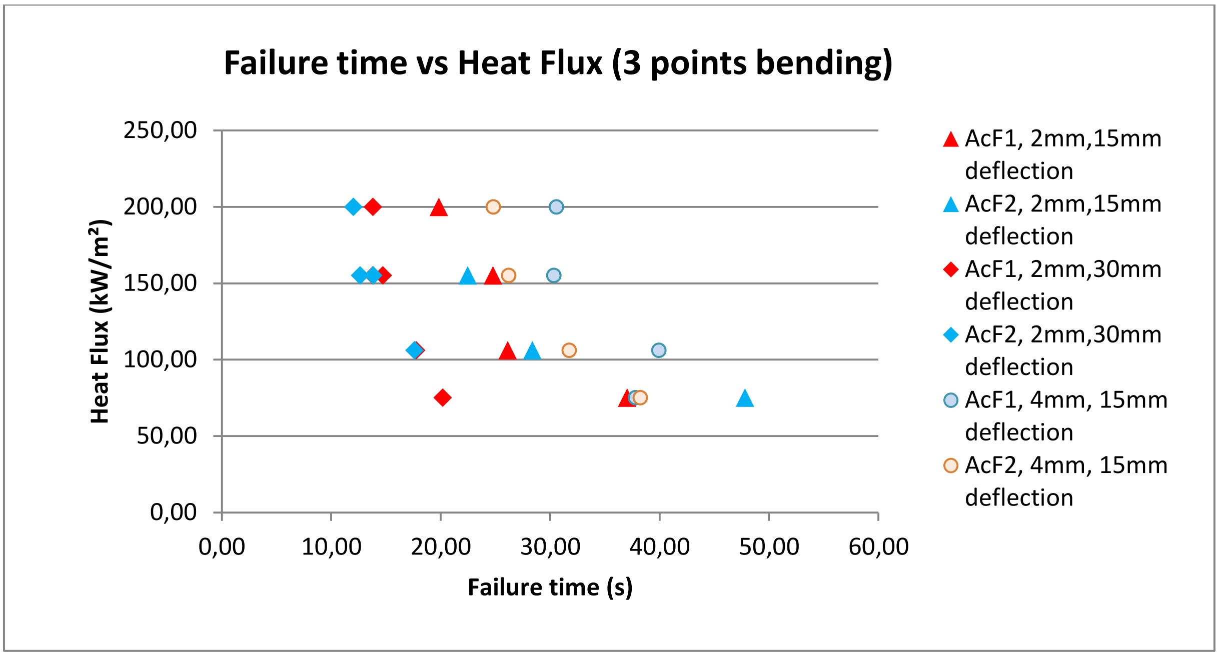

Figure 1 : Failure time for AcF1 and AcF2 at two different thicknesses exposed to heat flexes ranging from 75 to 100kW/m²

These results below give the outlines of AcF1 and AcF2 materials behavior exposed to fire aggression with the outer surface exposed to the flame in tension:

- Failure times observed in this tests for theses laminates are quite short, ranging roughly from 12 to 40 s, while the unloaded laminate is not burn-through after 1000s [3],
- For each deflection conditions, AcF1 and AcF2 behaviors for 2mm thickness are quite similar in terms of trends and failure times,
- 4mm thickness laminates exhibit similar behavior as the 2mm ones,
- For a given thickness, failure time increase as the stress decrease, as shown by failure time trend of 2mm thickness laminate vs. time, for 15 and 30mm deflection,
- Failure time increase with laminate thickness as shown by longer failure time for 4mm thickness laminate, 15mm deflection, comparatively to 2mm, 30mm deflection, while stresses in the outer surface are similar (296Mpa vs. 275Mpa).
- The observation of specimens broken with the specific stress field, tension for the outer surface exposed to the flame, shows pyrolysis and delamination of the plies exposed to the burner, as the rear side breaks in buckling mode, exhibiting kinking bands [4][5].

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5. Modeling and results

The objective of this work is to model the experiment using Abaqus® FEM software and Dakota optimization software to determine by inverse method and optimize thermal properties of composite material including pyrolysis effect. Based on back side specimen temperature measurement with different Heat Flux (75kW, 106 kW and 155kW), on specimens thicknesses of 2mm and 4mm, at mid span and 80 mm far, combination of all these temperature evolution have been used.

First, trial input data are managed by Abaqus® to find a solution of the problem; Dakota, thanks to a Python source code, pick the numerical curves and compare them with the experimental ones.
The difference between them is the residual function *u* that is minimized by the software, changing the variables which define the main function of the problem: the thermal conductivity and the specific heat.

$$u = \arg.\min\,(\mathrm{R})\ (\mathrm{eq.}\,2)$$

$$R = \left(\left(T_{num_1} - T_{\exp_1} \right)^2, \dots, \left(T_{num_n} - T_{\exp_n} \right)^2 \right)\ (eq.3)$$

The thermal model for the representation of the physical phenomenon is:

- two-dimensional
- bi-constituent (virgin material + char material)
- with thermal local equilibrium
- thermal properties function of the temperature
- heat flux incident constant
- take into account the change in density
- the assumption of ignoring the mass of volatile gases is done.

The Heat transfer governing equation, implemented in Abaqus® for calculating the temperature at any location of the composite is expressed as:

$$\rho(\mathrm{T})\mathrm{C_p}(\mathrm{T})\frac{\partial \mathrm{T}}{\partial \mathrm{t}} = \frac{\partial}{\partial \mathrm{x}}\left(\mathrm{k}(\mathrm{T})\frac{\partial \mathrm{T}}{\partial \mathrm{x}}\right)\ (eq4)$$

In this equation *T*, *t* and x, represent the temperature, time and distance below the fire-exposed surface. The C_p is the instantaneous specific heat and *k* thermal conductivity. The most significant effect in resin decomposition is the changing of thermal and mass properties of the material. For the evolution of density, the values are derived from TGA data on the composite at 10°C/min [6] (cf. Figure 2). In this case, most of the resin decomposition occurred in the range of 300° – 420° C.

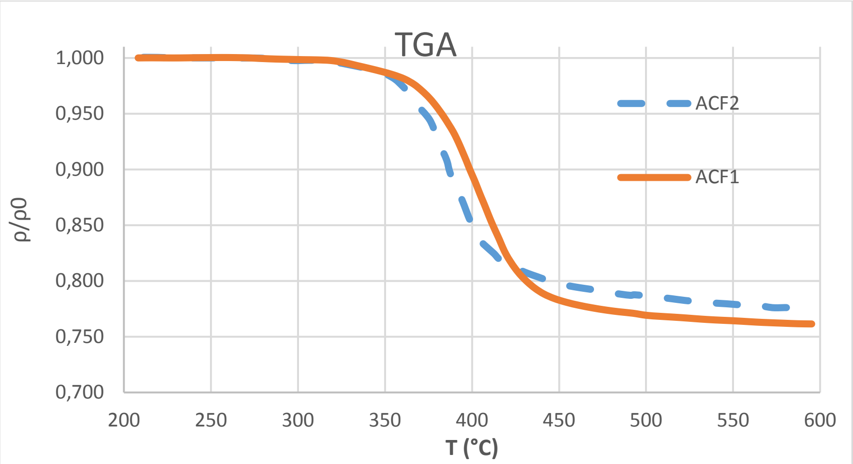

Figure 2 : Temperature-dependent values of density

Thermal conductivity and specific heat are determined using rule-of-mixtures:

$$k(T) = f_{virgin} * k_{char} + f_{char} * k_{char}\ \ (eq.5)$$

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$$C_p(T) = f_{virgin} * C_{p_{virgin}} + f_{char} * C_{p_{char}} \quad (eq.7)$$

The specific heat evolves with the temperature in accord with Henderson' model [7],

$$C_p = F * C_{p_v} + (1 - F) * C_{p_c} \quad (eq.8)$$

$$F = \frac{m - m_f}{m_0 - m_f} \quad ; \; C_{p_v} = C_{p_{v_1}} + C_{p_{v_2}} T \quad ; \quad C_{p_c} = C_{p_{c_1}} + C_{p_{c_2}} T \quad (Eq.8a;8b;8c)$$

- m_0 = initial mass
- m_f = final mass
- F = mass fraction of virgin material

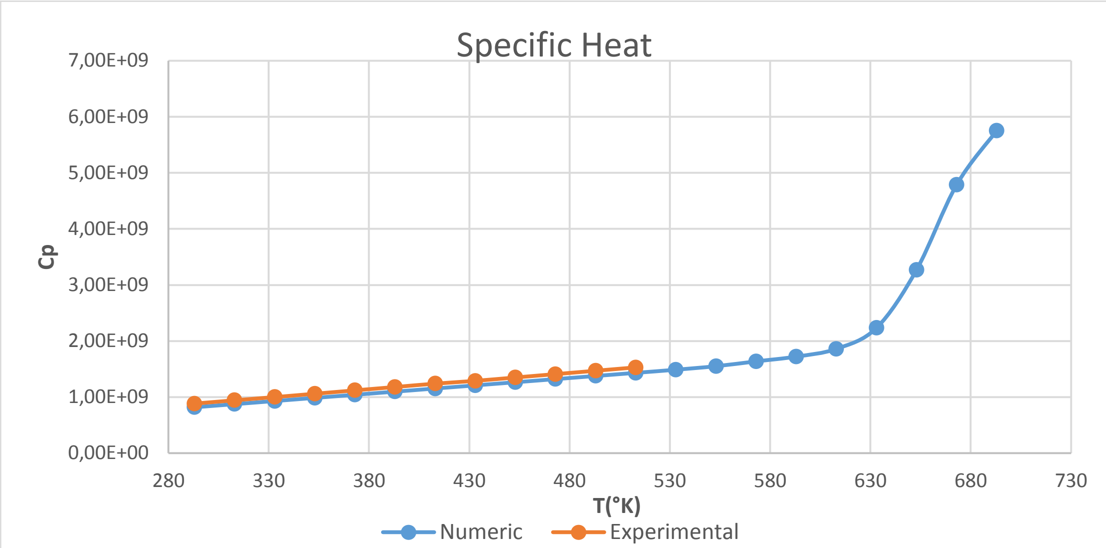

Figure 3 : Experimental vs. Numerical Cp by inverse problem

While thermal conductivity evolves in accord with Dimitrienko' model [8],

$$k(T) = \frac{k_{vo}}{1 - \varphi_{mo}} \left(\frac{T}{T_0}\right) (\varphi_m + n * \varphi_c) \quad (eq.9)$$

- k_{vo} = thermal conductivity at T_0
- φ_m = volume fraction of matrix
- φ_c = volume fraction of char
- φ_{mo} = volume fraction of matrix at T_0
- n = empirical constant

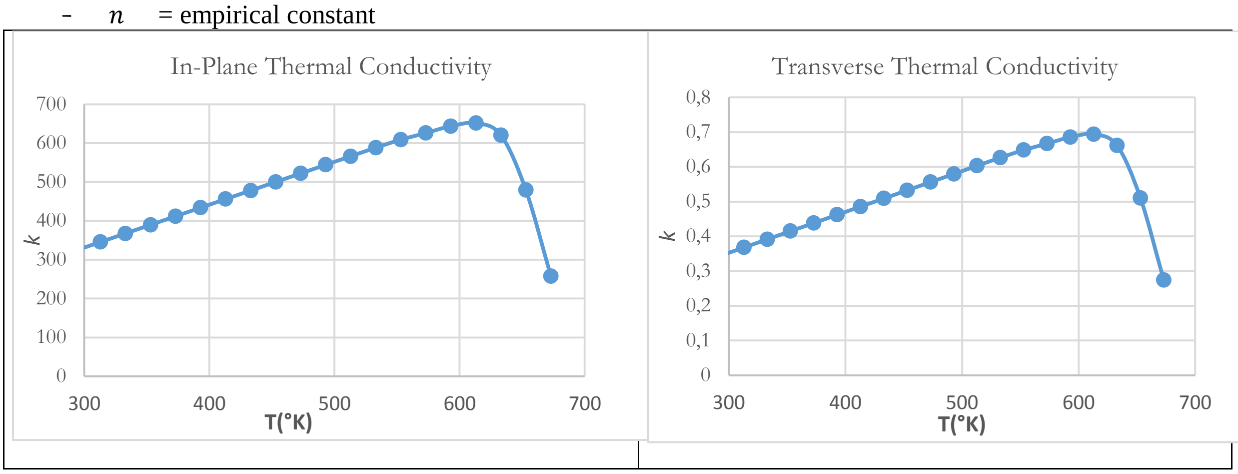

Figure 4 : Numerical Conductivity k

Another effect is simulated by the radiation emissivity (ε) which at high heat flux is modeled as a constant measured experimentally and fixed at 0,91.

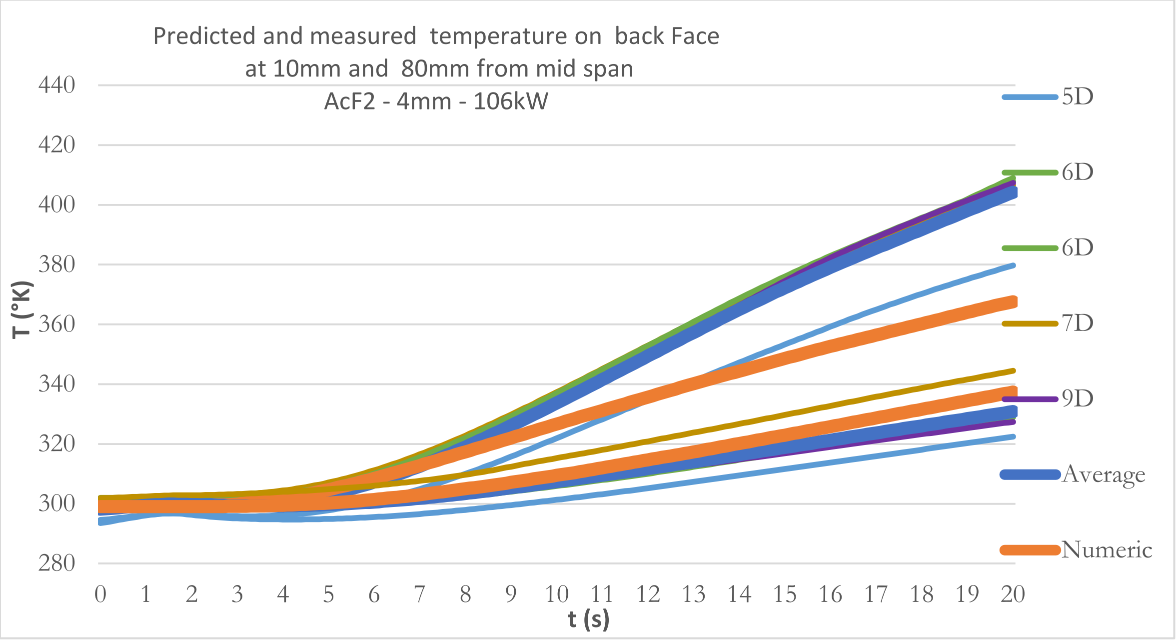

Figure 5 : Predicted and measured temperature at 10 and 80 from the centerline on the back face of 4mm thickness specimens AcF2 exposed to the burner at heat flux of 106kW

Figure 5 presents the comparison of the modeled and experimental back face temperature evolution at two different locations (10 and 80mm from mid span), for AcF2 laminate exposed at a flame of 106kW/m². As observed, the modeled temperature evolution fit very well temperature measured at 80mm from mid span while temperature at centerline is under estimated. Modeled temperature field were mapped and compared to tomography observation showing material damage in the bulk after 40s exposure and specifically matrix ablation of the heated surface (cf. Figure 6). Modeled damage zone shape is comparable to the experimental one in term of pyrolysis depth, while in plane pyrolysis area is slightly greater.

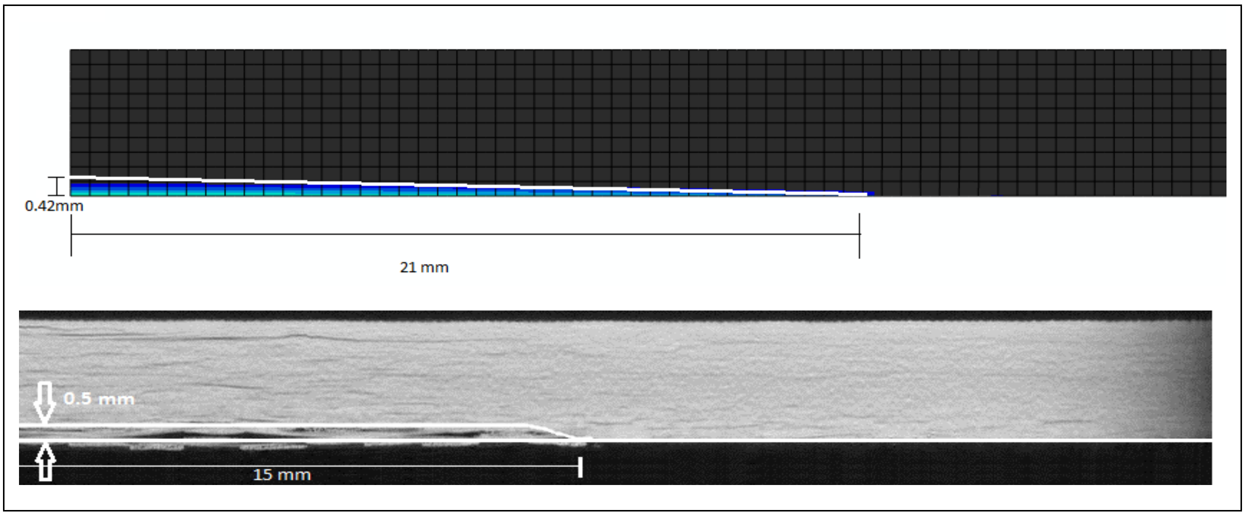

Figure 6 : Burner 106kW ACF2 4mm Pyrolysis Area after 40s exposure

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6. Conclusions

This paper has two main objectives:

- Evaluate impact of a load on carbon epoxy laminates under fire condition as it might occur in new composite structure aircrafts in case of post-crash fire or in flight fire,
- Modeling thermal behavior of carbon epoxy laminates using simple tools based on thermal transfer, taking into account evolution of specific heat, thermal conductivity and density as a function of temperature, in order to evaluate damage induced by thermal effects.

Tests on a small thermo-mechanical test bench have showed detrimental effect of an applied load on composite failure, leading to breakage in a very short time (12 to 40s), while the unloaded laminate is not burn-through after 1000s. These tests also put forward, dependence between failure time, heat flux, stress and laminate thickness.

A simple thermal model has been developed taking into account evolution of thermal properties and density with temperature determined by inverse problem and optimized with Dakota software. Prediction of temperature evolution on back face of specimen fits quite well with experimental temperature measurement and pyrolysis zone observed by tomography.

Further work will focus on mechanical modeling of the sample in order to predict the failure time.

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