

Infrared thermography for non-invasive characterization of thermal properties of CFR-Composite materials exposed to fire

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Abstract

The effect of fire exposure on the thermal behavior of carbon fiber reinforced (CFR) composite plates has been studied. Samples have been exposed to fire produced by a standard burner. The temperature evolution of both faces of the sample has been recorded by a two-camera system providing co-referenced and synchronized images. The camera imaging the "hot face" has been spectrally tuned in order to minimize the flame effect on the measurement. Several fire experiments have been performed with different exposure times. An adaptation for insulating materials of the classical flash method has been developed to obtain the values of thermal parameters for the burned sample. Different states of samples damage produced by fire application have been determined and characterized providing quantitative information about the thermal behavior of these materials exposed to fire.

1. Introduction

The use of fiber reinforced composites is growing rapidly in very disparate industrial sectors. The replacement of traditional materials by composites is due to the many advantages provided by the latter. As fiber reinforced composites combine high strength and rigidity with a light weight, tailorable properties and shape manufacturing, aerospace industry is the sector where these materials are becoming of increasing importance.

In aerospace industry, the fiber reinforced composite most widely used is a matrix epoxy resin in combination with carbon fiber [1]. Mechanical and thermal properties of different types of CFR composites present a strong dependence on temperature since, as temperature rises, agglutinant resins change state to liquid or vapor and carbon fibers become destroyed or carbonized. In spite of its importance, the effect of high temperatures on CFR composites is not well understood.

Infrared (IR) thermography is a well-established technique for temperature measurements, with many applications in the fields of predictive maintenance, non-destructive testing, etc [1]. As compared to standard methods based on the use of thermocouples, IR thermography has distinct advantages: it is non-invasive, provides two-dimensional rather than point measurements, offer a very fast response and the sample preparation requirements are comparatively simple [2].

A thermography study of CFR composites in fire application is presented in this paper. The thermal parameters of several burned and unburned samples have been determined experimentally. A acquisition system consisting of two IR cameras, one of them provided with a spectral selection to minimize the effect of fire in the infrared detection, has been used. This system has provided very valuable information on the structural damage produced by fire in the sample. The thermal parameters have been used to predict the thermal behaviour of materials under study. In order to achieve accurate values of these parameters, an adaptation to highly insulating materials of the classical flash methodology of Parker et al. [3] has been developed. The parameters obtained using this methodology have been used to feed a numerical analysis model of the fire experiments, reproducing very accurately the temperature evolution of the rear (cold) face of the plates knowing the hot face one (as measured with the front camera).

2. Work methodology

Several fire experiments have been performed in order to study, independently, the evolution of degradation states in the material and its influence on its thermal properties. For each experiment the analysis procedure has been as follows: first, the thermal parameters of the unburned sample are measured (pre-burning analysis). Then, the sample is exposed to a fire, established according to norm [4] during a prescribed time and once the burning has turned off and after reaching room temperature, the thermal parameters of the most affected area of the sample (the area where the flame impacted directly) are obtained (post-burning analysis).

All samples under study came from the same matrix, so the manufacture process has been the same in all the samples tested and, in the un-burned state, all of them have a flat shape, the same number of layers (nine) and same thermal properties previous to burn.

The pre-burning and post-burning analysis, and also the study of the temperature history of all the fire experiments, will provide the necessary quantitative information to understand the thermal behavior of the studied samples. Finally, all these data obtained will be enough to model CFR composites behavior under exposition to fire.

2.1. Thermal profiles dual system

A Thermal Profiles Dual System (TPDS) consisting of two time-synchronized and geo-referenced IR cameras has been designed and developed in order to correlate each opposite pixel on both faces (dual pixel) with both cameras. Their spectral range is the thermal infrared (8 to 14 microns wavelength). Each camera has been located to image on a sample side, thus the system measures simultaneously the temperature evolution of both faces during the burning process. The experimental setup of a burning test is shown in figure 1.



Fig. 1. Experimental setup of a burning test

The TDPS has demonstrated to be a powerful tool to study the thermal behavior of materials plates in fire test in an efficient way. Nevertheless one of the main problems arising in TDPS in-situ measurements is the high optical power impinging onto the camera located at the fire side (hot face) coming directly from the flame. It can easily saturate and even damage the detector or, alternatively, it is necessary to reduce dramatically its dynamic range, spoiling in both cases proper measurements. To avoid this restriction, this camera has been spectrally modified in order to minimize the high IR emission due to flame combustion gases and hot particles, taking advantage of the difference between the emission IR signatures of flame and surface plate [2]. In this way a proper signal can be obtained without losing temperature accuracy. The temperatures commonly provided by TPDS are brightness temperatures which are enough for the majority of the purposes nevertheless, sample emissivity can be introduced in the calculations in order to obtain real temperatures if necessary.

3. TPDS results and degradation states determination

TDPS can provide a variety of outputs from fire tests performed on a sample. Temperature images correlated on both sample faces as a function of time, temperature profiles and isotherms can be obtained. The temperature evolution of a specific pixel on both faces is a very useful indicator of the thermal behavior of the sample. For example, the temperature evolution of a dual pixel belonging to the area exposed to the flame in a 15 minutes fire test is shown in figure 2.

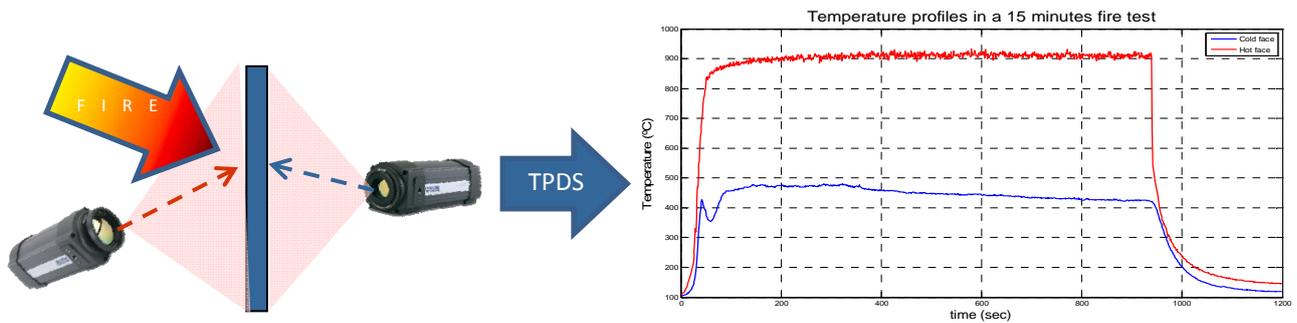


Fig. 2. Operation diagram and time evolution of a dual pixel as provided by TPDS

Figure 2 shows the temperature evolution during the fire test of a dual pixel. The pixel corresponding to the “hot face” where fire is applied appears with higher temperature, and the corresponding “cold” pixel (rear face) appears with lower temperature. The TPDS outputs make possible the detailed study of the thermal sample behavior. For example, different abrupt changes in the temperature time evolution on both faces under a constant fire load can be observed. The figure 3 shows it in more detail.

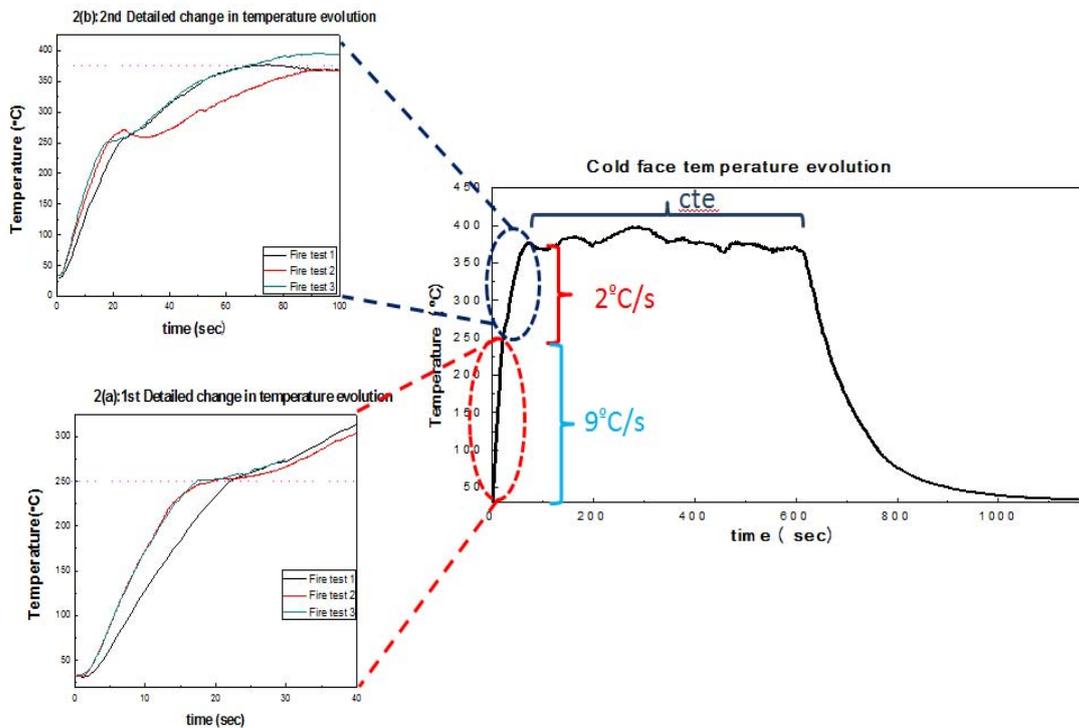


Fig. 3. Right: Example of temperature evolution obtained in a fire test of an area where the flame impacted directly. Left: show the detailed changes produced in different burnings, showing that the slope changes abruptly when a given temperature is reached whatever the sample and the rate of increase of temperature

In figure 3, abrupt slope changes in temperature evolution on the cold face can be observed in all fire tests. Every test has been carried out over unburned samples, according to the explained methodology. The results interpretation drives to the following conclusions.

Whatever the fire time exposure, the slope changes are produced always at the same temperature (see figure 2(a) and 2(b)). This result supports the hypothesis that these changes are produced by an abrupt change in the structural or compositional properties of the material with the temperature, when the resin disappears or the carbonization of the layers is reached. These internal effects produce a degradation state in the material altering the thermal properties in such a way that can be detected by abrupt slope changes in the surface temperature on both sides.

From the study of slope changes, two significant transition temperatures, one occurring approx at 250 °C and a second one at 375 °C have been found. These two temperature limits delimit three main degradation zones corresponding to each state in the surface: one for the regions that have rested always below 250 °C, the second one for regions whose temperatures have reached 250 °C but never exceeded 375 °C and the last one for those regions where temperature exceeded, at least once, 375 °C. These limits must correspond to irreversible changes in the inner structure or composition of the samples.

In order to understand better the thermal behavior of each degradation zone in the samples, three experiments has been carried out at different time fire exposure. Each time exposure has been defined in order to keep the sample under the limit temperature of each degradation state. In this way it is possible to analyze separately each of the three states. The programmed fire tests lasted respectively, 10 minutes (constant and almost zero slope), 80 seconds (slope =2°C/sec) and finally 30 seconds (slope=9°C/sec).

4. Experimental procedure for thermal parameters determination

It must be highlighted firstly the difficulty to obtain the thermal parameters of these materials by the classical methods, since they are very insulating materials. In addition, the thermal properties are very sensitive to and easily changed by the fabrication process. Moreover, if the samples have been degraded by direct fire application, as in this work, properties change in an unpredictable way.

In order to characterize the thermal properties of these materials a new method, at same time easy operational and accurate, has been developed. It consists of an adaptation of the classical flash method of Parker et al. [3] to very insulating materials, where the thermocouples are substituted by the IR cameras. Then, after emissivity correction, an accurate value of temperature is recovered and the thermal diffusivity obtained.

4.1 Adaptation of the flash method for the samples of interest

The classical flash method provides the thermal diffusivity of plane-parallel opaque samples with negligible heat losses [3]. A very short optical pulse emitted from a flash lamp is applied to the front face of the sample. The time evolution of temperature in the rear face is registered in order to calculate the diffusivity.

The flash lamp is used to eliminate the thermal contact resistance, while the heat losses are minimized making the measurements in a time short enough that very little cooling occurs [1]. Nevertheless this assumption cannot be held as true in the case of good insulating materials beyond a specific plate thickness, since the response in the rear surface would not be fast enough. Hence, the effect of convective cooling begins to be significant and must be taken into account in order to obtain an accurate value of thermal diffusivity. This is the case of the samples of interest in this work (CFR composites).

In our case and in order to determine cooling losses (conductive and convective) the normalized variable Nusselt number (Nu) has been defined in Eq (1).

$$Nu = \frac{L * h}{k} \tag{1}$$

Nu is a dimensionless ratio between convection and conduction. In Eq (1), L is the sample thickness, h the natural convection coefficient and k thermal conductivity. For large values of Nu , the maximum temperature reached by the rear surface after the flash, decreases significantly as compared to the lossless case, see figure 4. An accurate estimation of Nu will make possible to correct for the cooling losses in the flash response.

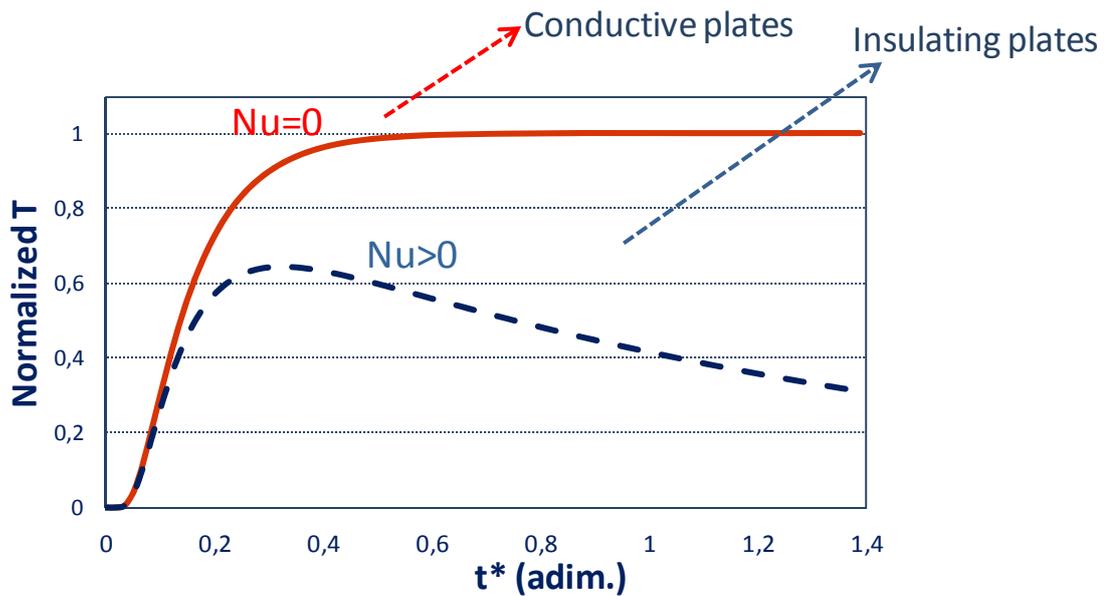


Fig. 4. Cooling losses effect on the flash response in the rear surface of the sample and the connection with the Nu value

In figure 4 a theoretical calculation of two different cases of flash response in the rear surface is shown. Dashed curve represents the response of an experiment without thermal losses, which is similar to conductive samples response with an appropriate thickness (fast material response). Typical response of the samples of interest where the convective losses cannot be avoided is plotted as continuous curve.

4.2 Determination of cooling effects. Fitting algorithms

An accurate value of Nu (for natural convection) is necessary to obtain an accurate value of thermal diffusivity in this kind of samples. To achieve this goal a fitting algorithm is used.

The fitting algorithm is based on finding the values of thermal diffusivity and Nu which provide the best fitting between the experimental and modelled response. Thus, thermal diffusivity and the natural convection parameter are obtained simultaneously, which is in fact a bonus of the method.

The experimental response is the graph of temperature response to the flash in the rear surface versus time, measured with an infrared camera $[T_{meas}(t)]$. Modeled response is defined by temperature versus time history obtained from a heat transmission model of the flash experiment $[T_{model}(\alpha, Nu, t)]$.

The algorithm seeks to minimize, using a maximum gradient method, the error function between experimental and modelled functions, calculated for all the measuring times t_i ; see Eq. 2. Besides, it chooses the initial values to optimize calculation time and to avoid falling into local minima which could provide an erroneous result.

$$\min \left[\sum_i [T_{model}(Nu, \alpha, t_i) - T_{meas}(t_i)]^2 \right] \Rightarrow (Nu, \alpha) \quad (2)$$

It is important to point out the different nature of the results provided by the algorithm. Thermal diffusivity is an intrinsic thermal parameter of the material; on the other hand Nu is a variable depending on the sample through L and k and the parameter h is an experiment dependent parameter. For this reason, Nu can change its value depending on room temperature, shape and position of the sample, and many other conditions.

Once Nu and α are calculated, the measured maximum temperature increment can be corrected for thermal losses and, once the flash energy density is known, an effective value for specific heat times density is calculated. Finally, this value, together with that for thermal diffusivity, provides the thermal conductivity of the sample. It is important to note that, if the sample zone under study is inhomogeneous, all the parameters obtained should be medium or effective values.

5. Results

Results of thermal parameters (thermal diffusivity, thermal conductivity and specific heat) obtained are shown in table 1.

Data presented in table 1 belong to the samples used in the three fire experiments described in section 3. For each sample pre and post burn analysis results are shown.

Table 1. Values of Thermal Parameters obtained with the proposed method are presented for each sample before and after fire application

Experiment description	Thermal diffusivity (m ² ·s ⁻¹)·10 ⁻⁷	Thermal conductivity (W·m ⁻¹ ·K ⁻¹)·10 ⁻¹	Specific heat (J·Kg ⁻¹ ·K ⁻¹)·10 ²
Unburned			
Sample used in burn of 30s	6.0	5.8	7.4
Sample used in burn of 80s	5.4	5.4	7.0
Sample used in burn of 10 min	6.2	6.2	7.6
Burned			
After burn of 30 s	1.2	1.1	6.7
After burn of 80 s	0.68	0.8	8.6
After burn of 10 min	0.49	0.5	8.8

In figure 5, thermal diffusivity dependence on burning time is presented. A strong variation can be observed in the first 50 seconds after fire application suggesting an important variation of the thermal properties of the samples in that short period.

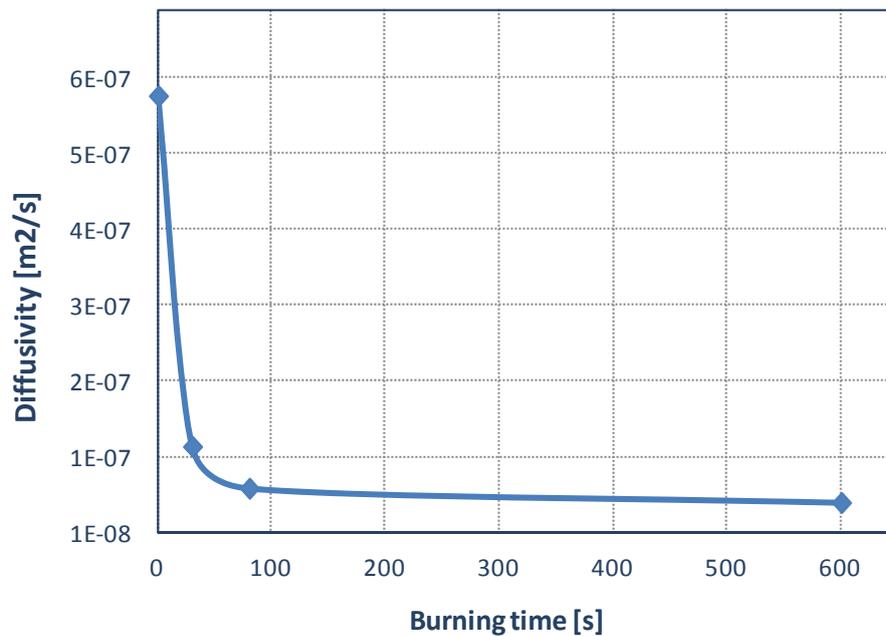


Fig. 5. Thermal diffusivity value as function as burning time. It drops a factor 10 in the first 50 seconds.

From the analysis of the whole data presented in table 1 and the curve of figure 5, it can be concluded that as fire time exposure increases, CFR composites change deeply their thermal properties. In fact, diffusivity and conductivity can change in a factor by 10 become more insulating and lowering the diffusivity, it suggests a structural change in the materials. All these results are summarized in figure 6.

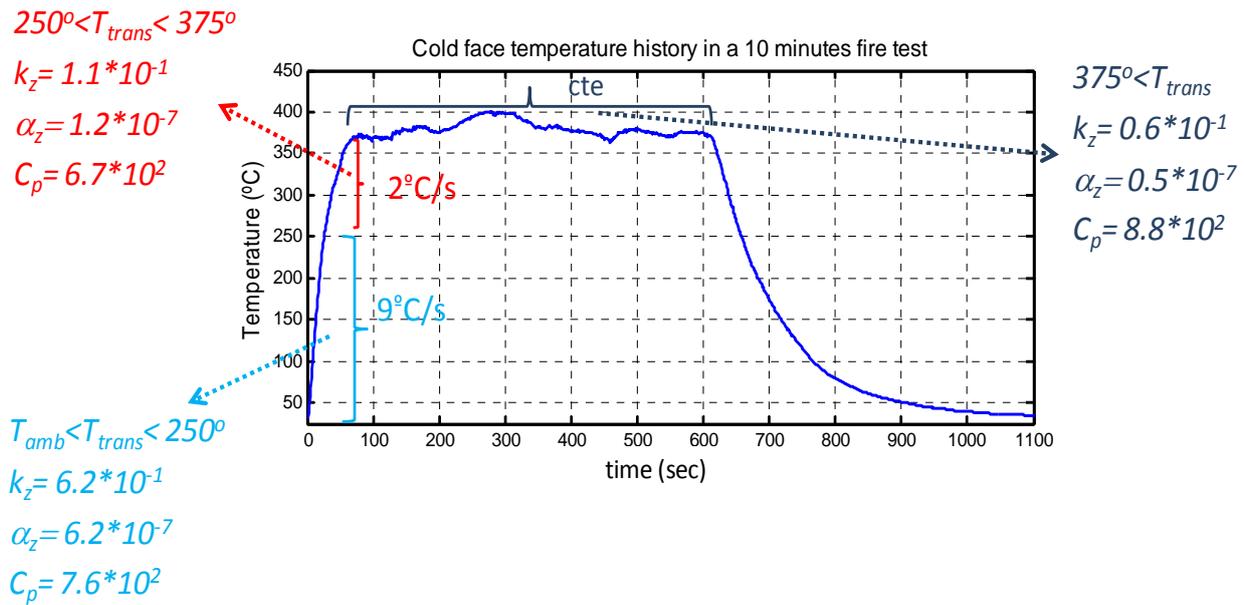


Fig. 6. Summary of results obtained with the proposed method, showing thermal parameters of each degradation state. Thermal diffusivity α , conductivity k and specific heat C_p (presented with international system units) of one of the samples can be seen for comparison purposes. A factor $\times 10$ can be reached after fire application.

6. Validation

In order to validate the results presented in figure 6, a computer model, based on a finite elements heat transfer, has been developed specifically for reproducing the performed experiments.

The input data to the model is the temperature evolution with time as measured with the IR camera during the experiment in the hot face. As a first approximation, thermal parameters in each finite element are kept constant up to crossing the transition temperature associated to each state, and are changed to the new parameters after crossing. Cooling effects (convection and radiation) of the specific validation experiment are previously unknown, for this reason they have been fitting keeping them constant all the time.

The validation procedure consists on the comparison between measured and modelled temperature time evolution in the rear face throughout the fire experiment. In this way the thermal behaviour of CFR composites exposed to fire is defined by obtaining their thermal parameters and validate through the temperature evolution in the cold face.

In figure 7 a result of the validation methodology is presented for a fire experiment, 10 minutes long, in the region under flame application. The temperature evolution in the hot face as measured (higher temperature line) has been used as the model input. The time evolution of measured temperature in the cold face is depicted as dotted line. Finally, the second line represents the time evolution of temperature in the rear surface obtained as an output of the model, fed with the thermal parameters. From the analysis of figure 7 a very good agreement between both curves can be seen.

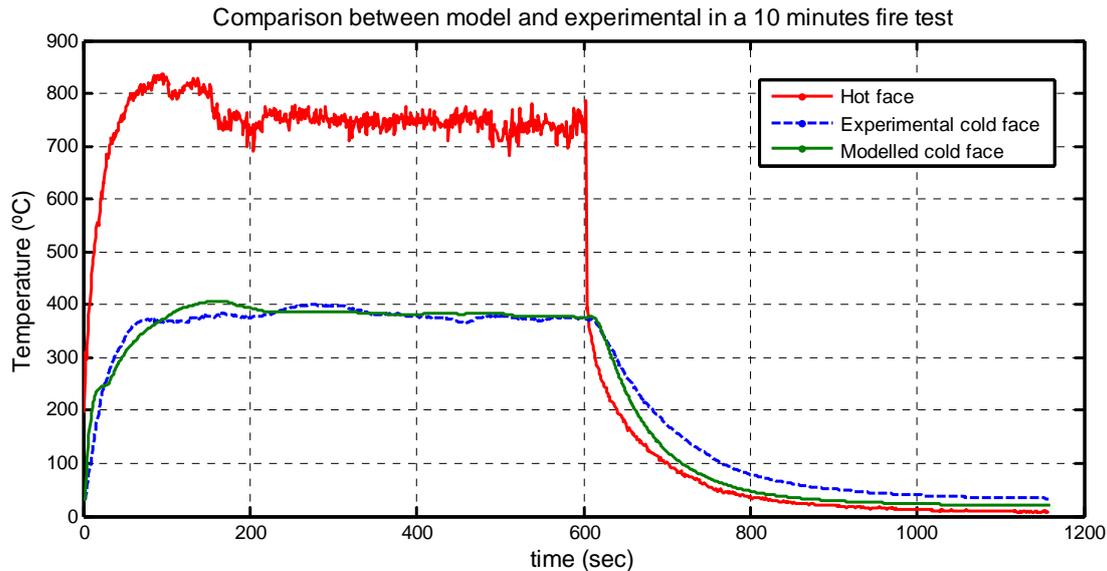


Fig. 7. Validation of the methodology proposed for a fire experiment 10 minutes long in a region under flame. Temperature evolution versus time in the cold face as measured (blue) and as obtained from the finite element model fed with the previously measured parameters (green). A very good agreement between both curves can be seen, showing the power of the proposed methodology. Red curve represents the hot face temperature, used as model input.

An error value, defined as the root mean square deviation between measured and modeled functions obtained through the validation methodology, has been calculated. For the whole experiment the root mean square deviation is 23.1 °C. The global deviation value can be considered as acceptable taking into account the temperature values reached in the fire experiment. This deviation value means that in spite of the differences found in the validation, none of them will be significant. Although, it must be noted there are some regions where deviation is perceptible (see figure 7; for example in cooling, after burner switch off). These deviations could come from different sources, as changes of thermal parameters and convection coefficients with temperature and other second order effects, to be studied in future works.

7. Summary and conclusions

An experimental procedure based on non-invasive infrared thermography to obtain the thermal parameters of plate samples of CFR composite materials has been developed. A new method adapting the classical Parker flash method to IR measurement for highly insulating plates has been proposed.

Fire experiments, established by norm, have been performed over CFR composite samples for aeronautic applications. In order to a proper comparison of the data obtained in the different experiments, samples coming from the same fabrication matrix have been tested, so the same pre-burning thermal parameters have been obtained.

Thermal diffusivity, thermal conductivity and specific heat has been retrieved from IR measurements for these samples, even for the different degradation states produced by the application of strong fire expositions. Nusselt number has been obtained too and used to take into account significant cooling effects produced in the application of flash method.

An objective way to determine the degradation state of the plates during the fire experiments, through the slope changes in the temperature evolution, has been proposed and validated.

In order to validate the obtained parameters they have been used as input to a heat transfer model. The output of this model matches to the empirical temperature data as measured on the cold face (23 °C deviation between modelled and measured data over 400 °C) The differences between both curves (empirical and modeled) must be assigned to second order effects like temperature dependence of the parameters or different ambient conditions in the experiments.

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