Thermal characterization of composite materials exposed to fire: quantitative comparison between classic and infrared-nondestructive methods

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Abstract
Several fire experiments have been performed using carbon fiber reinforced (CFR) composite plates exposed to fire produced by a standard burner. The temperature evolution of both faces of the plate has been recorded by a two infrared camera system providing spatially referenced and synchronized images. The camera imaging in the “hot face” has been spectrally tuned [1] in order to minimize the flame effect on the measurement. Different abrupt changes in the temperature time evolution on both faces under a constant fire load have been observed as a pattern. The abrupt temperature time evolution changes and abrupt degradation state changes during fire test have been connected [2]. In order to reinforce this link between degradation state and temperature evolution, studies at high temperatures using conventional techniques have been carried out [2]: thermogravimetry and differential scanning calorimetry.

1. Introduction
From the results attained in a previous study [3] an objective procedure to determine the degradation state of the plates during the fire experiments, through the slope changes in the temperature time evolution, was proposed and validated. It was proved that the changes in the temperature time evolution under load of fire are associated with a change in the thermal parameters those obtained since the adapted flash method [4] and validated by means of a comparison of standard finite element program with empirical results.

Classic thermo-analytical techniques have been and are now widely used and accepted in the characterization of materials. Thermal analysis includes all methods of measurement based on the change, with temperature, of a physical or mechanical property of the material. The manufacturing conditions of a product as well as its history and thermal treatments, are decisive in the final properties of the material, so the thermo-analytical techniques are essential in any process control over the manufacture of a material. The ability of these techniques to characterize quantitatively and qualitatively a variety of materials within a considerable range of temperatures has been crucial in the acceptance of these techniques.

In order to validate the IR-NDT techniques, proposed in this work, as a tool to determine the degradation states of the plates during the fire tests, the results are compared with those obtained from classic thermo-analytical techniques.

2. Methods
2.1 Thermal profile dual system (TPDS)

The TPDS has demonstrated to be a powerful tool to study the thermal behavior of materials plates in fire tests in a contactless approach. Nevertheless one of the main problems arising in TPDS in-situ measurements is the high optical power impinging onto the camera imaging the fire side (hot face) coming directly from the flame. 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

TPDS 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 point on both faces is a very useful indicator of the thermal behavior of the sample.

2.2 Classic methods: thermogravimetry and differential scanning calorimetry

The ability of these techniques to characterize quantitatively and qualitatively a variety of materials within a considerable range of temperatures has been crucial in its acceptance.

a. Thermogravimetry: Technique in which the mass of a sample is recorded vs. time or temperature.

b. Differential scanning calorimetry (DSC): It is based on associating the heating effects with chemical and phase transitions to a function of the temperature dependent reactions.

3. Results
3.1 IR-NDT
After several fire tests, all of them registered by infrared cameras, it is shown that the slope changes abruptly when a given temperature is reached, whatever the sample and the rate of increase of temperature [see Fig.1]. The results interpretation drives to the following conclusion: Whatever the fire time exposure, the slope changes are produced always at the same temperature (see Figs. 1(a) and 1(b)). This result supports the hypothesis that these changes are produced by an abrupt change in the structural or compositional properties of the material. Two significant transition temperatures have been found, one occurring around 250°C and a second one at 375°C.

![Figure 1](image1.png)

### 3.2 Classic methods

The results of the classic methods, Thermogravimetry (TG) [Fig.2] and Differential Scanning Calorimetry (DSC) [Fig.3], are shown below:

![Figure 2](image2.png)

![Figure 3](image3.png)

**4. Conclusions**

TG experiments show that separation with respect to the horizontal (temperature starting of mass loss) is equal to the 1\textsuperscript{st} temperature change by IR-NDT analysis (250°C) and the temperature of the first weight loss is equivalent to the 2\textsuperscript{nd} temperature (375°C), see Fig.2. Similarly, DSC experiments also show heat flux changes in the sample during heating at these same temperatures (see Fig.3). Results from classic methods concluded that the temperatures of degradation state change match with those obtained through IR-NDT analysis. Moreover, it has been observed that independently the heating rate, the degradation state changes are associated uniquely with temperature. These results connect the different standard techniques with IR-NDT methods reinforcing its validity as a tool to determine the degradation state of the plates during the fire experiments.

### References


