International Journal of Thermal Sciences 112 (2017) 427–438

Contents lists available at ScienceDirect

International Journal of Thermal Sciences

journal homepage: www.elsevier.com/locate/ijts

Surface temperature of carbon composite samples during thermal degradation

Zoubir Acem [a, b], Damien Brissinger [a, b], Anthony Collin [a, b], Gilles Parent [a, b], Pascal Boulet [a, b, *], Thi Hay Yen Quach [c], Benjamin Batiot [c], Franck Richard [c], Thomas Rogaume [c]

[a] Université de Lorraine, LEMTA, UMR 7563, Vandoeuvre-lès-Nancy, F-54500, France
[b] CNRS, LEMTA, UMR 7563, Vandoeuvre-lès-Nancy, F-54500, France
[c] PPRIME Institute, CNRS, Université de Poitiers, ISAE ENSMA, BP 40109, F86961, Futuroscope cedex, France

ARTICLE INFO

Article history:
Received 15 September 2016
Received in revised form
11 October 2016
Accepted 7 November 2016
Available online 13 November 2016

Keywords:
Pyrolysis
Cone calorimeter
Infrared imaging
Radiative transfer
Temperature identification
Heat flux
Flame radiation

ABSTRACT

An infrared camera and a Fourier transform infrared spectrometer were used simultaneously to observe the radiation from the surface of carbon composite samples during thermal degradation experiments. Surface temperatures were estimated from radiation measurements conducted during cone calorimeter tests. Infrared spectra and images were post-processed involving a subtraction method between successive images, to withdraw the high incident flux from the cone calorimeter partly reflected by the sample. The surface intensities were first evaluated as a function of time. Then, an identification step was implemented linking the rise in intensity between two time steps with the increase in sample temperature. Corrections were introduced for the reflection of the incident radiation from the heater and for the true emissivity of the material. Both experimental devices – camera and spectrometer – showed temperature evolutions in a good agreement. The sharp temperature increase of the sample and the auto-ignition of the pyrolysis gases on the surface were observed and evaluated for various incident fluxes from the cone between 20 and 65 kW/m^2. Measurements carried out with a short time step down to 0.2 s further allowed an evaluation of the supplementary flux due to the auto-ignition with a peak up to 20 kW/m^2 for an incident flux of 35 kW/m^2 on a carbon composite sample with surface 50 mm × 300 mm and thickness 5 mm.

1. Introduction

The study of the thermal degradation of materials provides some of the input parameters required for the prediction of their ignition and combustion. Standard characterization involves samples submitted to given external radiative fluxes, usually produced by a high temperature lamp in the Fire Propagation Apparatus [1], or a coil when using a cone calorimeter [2]. A calibration with a heat flux gauge ensures that the incident flux is well controlled and data are registered for the mass loss experienced by the sample as a function of time. This problem involves a complex coupling of heat and mass transfer. Therefore, temperature data are also required for a better understanding of the phenomena and for validation purpose. These data are then compared to results obtained with degradation and combustion models, which are needed in fire simulations for prescribing the mass loss rate and for predicting the heat release or the heat transfer inside the materials. The knowledge of the sample surface temperature is of particular interest for the description of the heat transfer inside the sample and for the definition of reliable boundary conditions. Thermocouples may be used to measure this surface temperature, but the confidence in the acquired data is questionable as direct radiation to the thermocouple may occur from the source or from the flame developing on the surface after sufficient heating. In addition, this is a high temperature measurement possibly disturbed by a non perfect contact (a specific problem which would be encountered in the present study devoted to a carbon composite material with a low conductivity) or also because of soot deposit. To avoid these problems,

* Corresponding author. Université de Lorraine, LEMTA, UMR 7563, Vandoeuvre-lès-Nancy, F-54500, France.
E-mail address: pascal.boulet@univ-lorraine.fr (P. Boulet).

http://dx.doi.org/10.1016/j.ijthermalsci.2016.11.007
1290-0729/

measurements inside the sample can be done with embedded thermocouples, extrapolating the values to the surface. Stoliarov et al. [3] presented temperature data measured with thermocouples embedded as close as possible to the surface (1.5 mm below it) in order to characterize the top layer temperature. Pizzo et al. [4] on PMMA and Hidalgo et al. [5] on a carbon composite sample from the same series as the one studied here, to name some recent studies, presented temperature measurements with embedded thermocouples at various positions inside the materials. Such measurements are usually used as validation data for models. Temperature identification could be also sought based on the observation of the back surface, which is protected from direct irradiation. However, other difficulties are raised owing to the non-stationary conditions and to the problem complexity (including the required model for heat transfer, through a complex medium with varying properties and the difficulty to do the measurement itself). Li et al. carried out such measurements at the back face and examined the accuracy of the surface temperature [6,7]. Data were then used for pyrolysis model validation purpose.

The present work was conducted to test another method based on the observation of the flux emitted by the front surface with a Fourier Transform InfraRed (FTIR) spectrometer and an infrared (IR) camera. Our objectives were twofold: (i) to observe the radiation sent by the irradiated surface during a cone calorimeter experiment in order to follow the temperature increase and to check for the temperature homogeneity on the surface; (ii) to develop a reliable method to identify the temperature despite the difficulty raised by the experimental setup (limited accessibility and strong background irradiation because of the cone itself). IR thermography was already used in the past to observe solid surfaces through a flame. For example, De Vries et al. [8] evaluated surface temperatures in rack-storage fires with a long wave camera (expecting limited perturbations by smoke and particles). They observed perturbations in the measurement because of the flame, evaluating the related uncertainty in the measured temperature to approximately 50 K (in a case of a large scale fire). Melendez et al. [9] also measured surface temperature in the case of a plate heated by a burner flame. They used a combination of an optical filter and an image processing involving the flame image subtraction to withdraw the perturbation by the flame. Similar ideas were used here, but in different conditions because the heat source was not a burner and the flame ignition or extinction could not be controlled.

The FTIR spectrometer and the IR camera were already used by present authors on several applications related to fire [10–13], for the characterization of sources or flames in particular. Intensities from flames and radiative sources were especially measured in various situations. Based on past experiences [14,15], spectra can be analyzed to identify characteristic temperatures in some wavenumber ranges. Filters can be also added to the camera in order to limit the perturbation of the signal by the flame and to isolate the surface radiation, which is then analyzed to identify its temperature. These methods were used here when registering the radiation from the sample surface.

For the present study, composite carbon samples were degraded under a cone calorimeter, varying the incident heat flux between 20 and 65 kW/m^2. The surface emission of the samples was simultaneously registered with the spectrometer and the camera. Then, several identification methods were tested in order to obtain the temperature and a satisfactory confidence in its time-varying evolution. In the following sections, the experimental setup will be described first. Then, the radiative transfer model and the identification methods will be presented. Finally, results will be discussed for the evolution of the surface temperature of the carbon composite.

2. Experimental setup

2.1. The composite sample

The material studied in the present work is made of carbon fibers in an epoxy matrix, a composite used for high pressure hydrogen storage tanks. Samples were prepared in the frame of the FIRECOMP European project aimed at characterizing the degradation of such materials when submitted to fire sources. The composite is mainly composed of carbon (85% of the total weight according to the available elementary analysis conducted in the frame of the project [16]). Specimens were cut in large composite cylinders, providing quasi-flat samples for degradation tests under cone calorimeter.

2.2. The cone calorimeter combined with infrared metrology

A picture of the setup is presented in Fig. 1. In this case, a standard cone calorimeter [1] was used in vertical configuration for the degradation of a 100 mm × 100 mm x 5 mm sample. The composite sample was placed in a sample holder specially developed for this application. This one was manufactured into a block of aluminum, and a cover was used in order to maintain the sample in the right position. The opened surface of the cover was 88.4 cm^2 in accordance with ISO 5660 [1]. It is important to note that no insulation was used in the back surface of the sample. The heat source was in fact a truncated conical heater and the coil of the cone can be seen in the picture. Radiation was provided from this coil toward the sample surface, leading to its thermal degradation. The picture was taken after auto-ignition of the gas mixing as shown by the flame developing along the sample surface. The observation in the infrared range was done along a path centered on the cone axis through its aperture, using a camera and a spectrometer which are described below. A silicon beam splitter divided the radiation signal toward the infrared camera and the spectrometer for a simultaneous observation of the same area with both devices. A typical test started when the sample was suddenly submitted to the radiation emitted by the cone at $t = 0$ s (removing a shield used between the heater and the sample to allow the heating of the cone up to the stationary regime while protecting the sample before the experiment starts). Four different incident fluxes were considered: 20, 35, 50 and 65 kW/m^2 (values obtained from heat flux gauge measurements carried out at the sample position in standard use of the cone [1]). Experiments were repeated twice to check the repeatability.

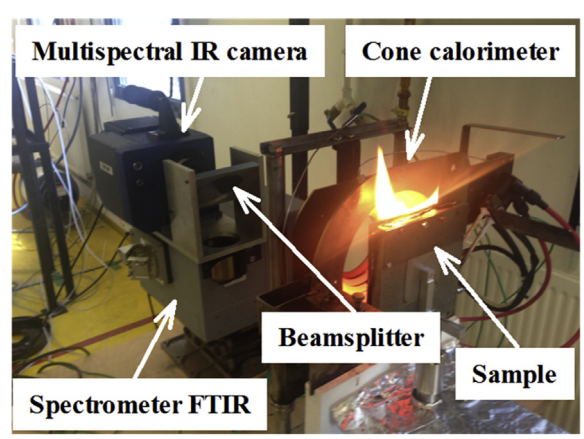

Fig. 1. Picture of the setup with the cone calorimeter, the spectrometer and the IR camera.