



Identification of a surface temperature during the thermal degradation of a carbon composite sample.

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Abstract : Surface temperatures were estimated on carbon composite samples during thermal degradation experiments using a cone calorimeter. The method was based on data acquisition in the infrared with a spectrometer and a camera. Infrared spectra and images are affected by the progressive temperature increase and the appearance of the pyrolysis products or the ignition of the gas mixing on the surface. This non intrusive observation method is therefore a powerful tool for a quantitative evaluation of the complex transfers at the surface, provided a post-processing of the signal can be performed carefully. 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 by the sample and for the true emissivity of the material. Results showed a sharp increase during the first instants up to several hundreds of kelvins per minutes, confirmed by both devices (camera and spectrometer independently).

Key words : fire propagation, radiative transfer, temperature identification.

1. Introduction

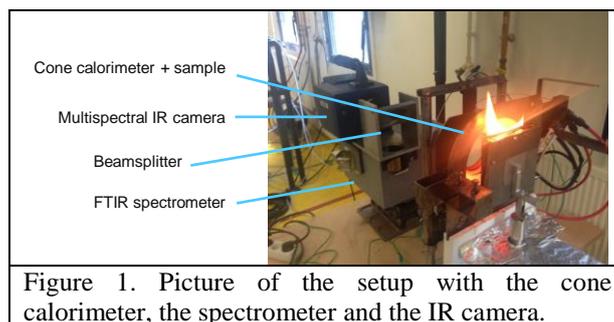
The study of the thermal degradation of materials provides some of the input parameters required for the prediction of their ignition and combustion. Samples are submitted to given external radiative fluxes, produced for example by a high temperature lamp in a Fire Propagation Apparatus, or a coil when using a cone calorimeter [1-2]. A calibration ensures that the incident flux is well controlled and data are registered for the mass loss or for reference temperatures. These data are needed for the degradation and combustion models when prescribing the mass loss rate and 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 or for the model validation. 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 and because this is a high temperature measurement disturbed by a non perfect contact (especially with the composite of interest in the present study) or also by the flame developing on the surface after sufficient heating. To avoid this problem, measurements inside the sample can be done with extrapolation to the surface, or identification can be sought based on sample rear surface observation. However, other difficulties are raised owing to the non-stationary conditions and to the problem complexity (including the difficulty to do the measurement itself). The present work was therefore 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 camera. These devices were already used by present authors on several applications related to fire applications [3-6], for the characterization of sources or flames in particular. For the present study, composite carbon samples were degraded under a cone calorimeter varying the incident flux conditions. The surface emission was registered with the spectrometer and the camera simultaneously. 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 method will be presented. Finally, results will be discussed for the evolution of the carbon composite surface temperature.

2. Experimental setup

2.1. The cone calorimeter combined with an infrared analysis

A picture of the setup is presented in Figure 1. A standard cone calorimeter is used in vertical configuration for the degradation of a 100mm x 100mm x 5mm sample. The heat source is in fact a truncated conical heater and the coil of the cone is visible on the picture. This latter was taken after ignition of the gas mixing as shown by the flame developing along the sample surface. The observation in the infrared range is done along a path centered on the cone axis through its aperture. A silica beam splitter divides the radiation signal toward the infrared camera (Orion SC7000 by FLIR) and the spectrometer (IRCube by Bruker) for a simultaneous observation of the same area. The camera can be used with filters in order to isolate images at given wavenumbers. For the present work, the filter at 2564 cm^{-1} was especially used, corresponding to a spectral region where gases do not contribute to the radiation, hence preventing from any perturbation of the image processing by possible combustion gases. On the contrary, the spectrometer provided results over the whole spectral range between 800 and 5000 cm^{-1} in the present study. A typical test started when the sample was suddenly submitted to the radiation emitted by the cone at $t = 0\text{ s}$ (removing a shield used between the heater and the sample to allow the heating of the cone up to the stationary regime while shielding 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.



2.2. Experimental data

Figure 2 and 3 show typical spectra registered during experiments conducted with incident fluxes equal to 35 and 50 kW/m^2 respectively. Data were converted into equivalent intensities after comparison with calibration data obtained using a high temperature blackbody (M330 by Mikron, at 1500°C). Spectral intensities are plotted as a function of wavenumber for different times. The intensity level increases with time as a consequence of the surface temperature increase. For incident flux equal to 35 kW/m^2 (Figure 2) spontaneous ignition was not observed and the spectrum shape was due to emission by the surface affected by the sample emissivity, with some sharp variations in definite bands due to the gas influence (mainly absorption by atmospheric water vapor and carbon dioxide). For an incident flux equal to 50 kW/m^2 (Figure 3), the first increasing phase was followed by a sudden increase in the intensity level and a simultaneous sharp peak observed around 2300 cm^{-1} as a consequence of a flame suddenly developing along the surface. Then, a further increase was observed before a decreasing phase as most of the sample was degraded. These four phases are presented in the Figure schematically. Camera images were post-processed in a similar way (two examples are given in Figures 4 and 5), mainly in order to investigate the homogeneity of the radiation emitted by the surface. The two images are presented in terms of intensity after a conversion of the camera signal based on reference data obtained in front of our reference blackbody. The surface temperature is then identified based on the intensity, as described in the next section. Note that the white circle at the centre of the image features the area observed by the spectrometer (which receives radiation in a limited solid angle and does not see the whole plate as the camera does). All data processing in the following sections (based on camera or spectrometer data) correspond to averages computed for the area inside this circle, with diameter 2.3 cm. On the images, some heterogeneities are seen, which

correspond to the carbon fiber orientation, but the temperature is not much varying along the surface. Figure 5 shows some hot points which probably correspond to the flame developing along the surface in this case.

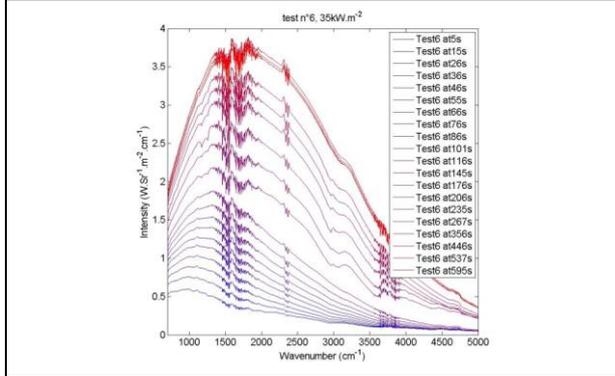


Figure 2. Radiative intensity emitted and reflected by the sample surface at different times when irradiated by a cone calorimeter with a 35 kW/m² flux.

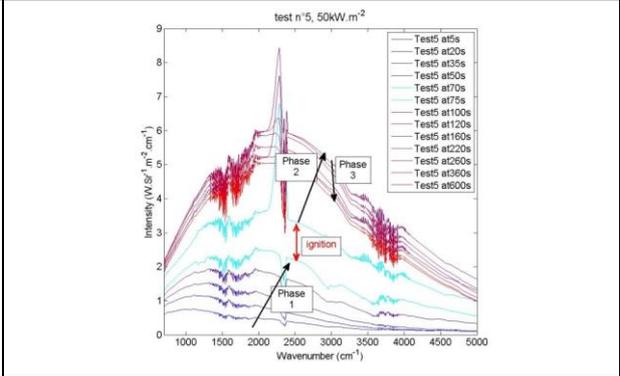


Figure 3. Radiative intensity emitted and reflected by the sample surface at different times when irradiated by a cone calorimeter with a 50 kW/m² flux.

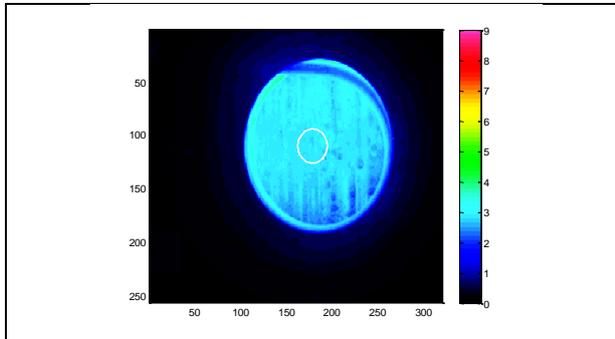


Figure 4. IR image at 2564 cm⁻¹, converted in intensity. Incident flux 35 kW/m², time t = 390s

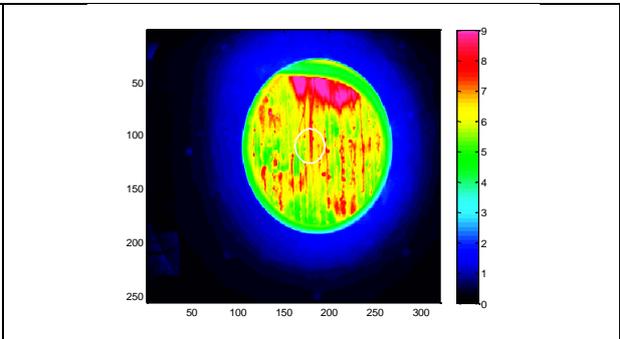


Figure 5. IR image at 2564 cm⁻¹, converted in intensity. Incident flux 50 kW/m², time t = 80s.

3. Model for temperature identification

3.1. Radiation model

The basic idea is of course to use the link between the temperature and the intensity emitted by a surface, written using the Planck's law [7] for a black surface :

$$I_{b\eta} = \frac{1}{\pi} \frac{C_1 \eta^3}{e^{\frac{C_2 \eta}{T}} - 1} \quad (1)$$

where $C_1 = 3.7418 \cdot 10^{-16} \text{ W.m}^2$, $C_2 = 1.4388 \cdot 10^{-2} \text{ m.K}$ and η stands for the wavenumber in m⁻¹.

The measured intensity is then expected to allow identifying the surface temperature, simply inverting relationship (1). However, the received flux is in fact a combination of the flux emitted by the surface, plus the reflection of the radiation coming from the cone calorimeter. Hence, the measured intensity corresponds to:

$$I_\eta = \varepsilon_\eta I_{b\eta}(T(t)) + \rho_\eta I_{b\eta}(T_{cone}) \quad (2)$$

with $T(t)$ standing for the surface temperature, T_{cone} for the cone temperature, ε_η is the spectral emissivity and ρ_η is the surface reflectivity.

This assumes a lambertian irradiation from the cone (which is close to a blackbody) and a diffuse reflection. This reflection part is even the major contributor to the radiation received from the surface during the first instants of the experiment (note that radiation from the surroundings at ambient temperature is clearly negligible as compared to the cone radiation, it is not included in this relationship). Reflection cannot be simply removed after a rough evaluation of the incident flux, because any uncertainty in this value or in the reflectivity would result in strong errors in the temperature during this first step, when the surface temperature is still moderate. In the second step of the temperature evolution, the emission becomes more and more influent in the global signal as

the surface temperature increases. Then, the uncertainty due to the reflection decreases. Finally, after ignition along the sample surface, the flame also provides an additional flux directly emitted toward the spectrometer or reflected by the sample surface, which must be taken into account.

For the present analysis, these parasitic reflections were accounted for in the signal processing thanks to an image subtraction method. It assumes that no significant change occurred in the incident radiation (and thus in the reflected intensity) during the experiment, since the main part was due to the cone calorimeter which was in stationary regime. The subtraction eliminates the reflection contribution and the variation in intensity is correlated to the temperature increase. The method can be summarized as follows:

- i. Evaluation of the instantaneous intensity from the direct signal acquisition thanks to the calibration data obtained with the high temperature blackbody
- ii. Calculation of the difference between the measured instantaneous intensity and the reference value at initial time (meaning the subtraction of the reflection from the cone) : $\Delta I_{\eta 0} = I_{\eta}(t) - I_{\eta}(t = 0)$
- iii. Evaluation of the temperature $T(t)$ from an inversion based on the Planck's law applied to the intensities involved in step (ii):

$$T(t) \approx \left[-\frac{1}{\eta c_2} \ln \left(\frac{\pi \Delta I_{\eta 0}}{\varepsilon_{\eta} c_1 \eta^3} + e^{-\frac{c_2 \eta}{T(t=0)}} \right) \right]^{-1} \quad (3)$$

The temperature evaluation is initialized at $T(t = 0s) = 20^{\circ}\text{C}$.

The possible drawback of this method is that the reflection subtraction assumes no change during all the experiment, which is questionable especially when the flame appears along the sample. Therefore a second method was tested, replacing step (ii) by a subtraction between two successive images, such that the reflection part is only assumed constant between two acquisition times (down to 1s for the camera, around 3s for the spectrometer). The method becomes:

- i. Evaluation of the intensity from the direct signal acquisition thanks to the calibration data obtained with the high temperature blackbody
- ii. Calculation of the intensity difference between two successive acquisitions $\Delta I_{\eta} = I_{\eta}(t) - I_{\eta}(t - \Delta t)$
- iii. Evaluation of the temperature $T(t)$ from the inversion of the Planck's law for radiation:

$$T(t) \approx \left[-\frac{1}{\eta c_2} \ln \left(\frac{\pi \Delta I_{\eta}}{\varepsilon_{\eta} c_1 \eta^3} + e^{-\frac{c_2 \eta}{T(t-\Delta t)}} \right) \right]^{-1} \quad (4)$$

Initialization is still done with $T(t = 0s) = 20^{\circ}\text{C}$ and an iterative process is used. The reflection correction is better, but any error in a temperature evaluation at time t is kept and cumulated with other possible errors during all the experiment, which could cause some deviations for the final result. Actually, both methods gave the same results in our case and the two devices (spectrometer and camera) also gave the same temperature evolutions (within a reasonable uncertainty range) as it will be seen later in the section results. This shows that the reflection correction is well controlled and thus gives some confidence in the results.

Note that the surface emissivity has to be known prior to the data analysis. A preliminary evaluation of the radiative properties of the surface was conducted measuring the spectral reflectivity and transmissivity of the sample surface, as presented in [6] for example. It was found that the sample was opaque (zero transmissivity) with a near gray behavior with reflectivity 0.09 and corresponding emissivity 0.91 in the spectral range around 2564 cm^{-1} .

3.2. Temperature identification and sensitivity analysis

Before using the method seen in the previous paragraph on real experimental data, a numerical test was carried out first, in order to check our ability to identify a given temperature evolution in an accurate manner. A typical evolution of the temperature was considered during 300s in the range between 293 K and 800 K according to a representative exponential law such as $T(t) = T(0) \cdot \left[1 - e^{-t/\tau} \right]$, with $T(0) = 293 \text{ K}$ and $\tau = 100 \text{ s}$. This relationship was chosen as it looks like the temperature increase observed in present degradation tests, as will be seen later. Then, the theoretical intensity sent by the surface was predicted using relationship (2), considering an emissivity close to the actual value ($\varepsilon_{\eta} = 0.90$) and diffuse reflection (with reflectivity $\rho_{\eta} = 0.10$) of an incident intensity coming from a blackbody at temperature $T_{cone} = 992 \text{ K}$ (the set temperature of the cone for an incident flux of 35 kW/m^2 in our experiments). This ‘‘synthetic’’ intensity was then used for a mock test of temperature evaluation from intensity data. This test is a kind of inverse crime, since the same model is used in a forward and a reverse application for the data generation and the identification. Therefore, it cannot be considered as a full validation. However, it allows investigating the sensitivity of the results to a hypothetic error in the emissivity (value 0.85 tested), in the initialization (error tested in initial temperature 303 K instead of 293 K), or due to a noisy acquisition (intensity acquired with a random noise up to 5% of the signal). Results are presented in Figure 6 for the different temperature evaluations. Only the results based on the subtraction of the data obtained at two

successive steps are presented, as no difference could be seen between the results based on relationships (3) or (4). All curves (basic evaluation and data disturbed by error in initial temperature, emissivity or noise) seem to overlap and there is no significant discrepancy or uncertainty visible with this temperature scale. Discrepancies in the evaluation appear when the absolute error (true temperature minus identified temperature) is plotted as a function of time (Figure 7). Evaluation at the first instants may present uncertainties if initial conditions are not well known, but results converge toward the same values for larger times, which are very close to the exact result if the emissivity is well known. In this case, long time evaluation can be taken with confidence, error being less than 1.5 K for an absolute temperature of 800 K. If an error occurs in the emissivity evaluation, the temperature may shift from the exact value, but the discrepancy is still in a range of 10 K here, as compared to the 800 K of the exact value. As a consequence, present results are obtained with moderate or weak uncertainty. This is especially due to the fact that the possible error due to reflection is well controlled thanks to the image subtraction process. Without such a method, approximation in the reflection evaluation would result in dramatic uncertainties during the first instants.

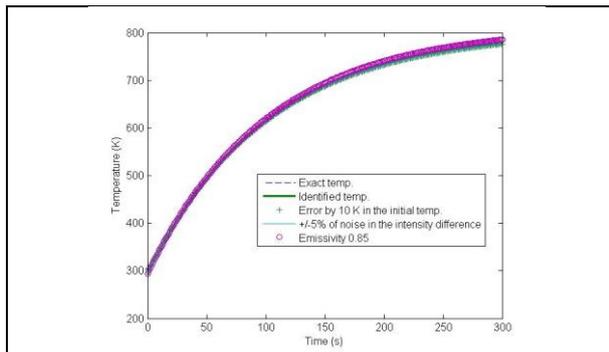


Figure 6. Numerical test on the surface temperature evaluation. Exact temperature (dotted line) compared with identified data introducing possible measurement uncertainties (noise, error in emissivity and in temperature acquisition).

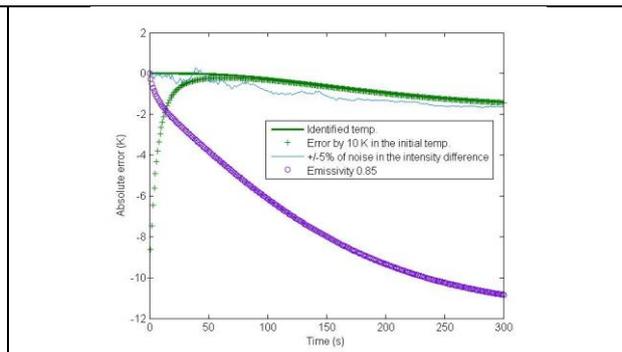


Figure 7. Absolute error corresponding to the results presented in Figure 6.

4. Results and discussion

As above-explained, the present method was used for the study of carbon composite samples under incident fluxes equal to 20, 35, 50 and 65 kW/m². The corresponding temperature evolutions with time are presented in Figures 8 and 9. Figure 8 gives an idea of the confidence that could be given to the results for two chosen fluxes (20 and 50 kW/m²). First of all, tests were repeated twice in order to check the repeatability (see the two sets of data per fluxes very close one from the other). Then, the two above-discussed methods, namely reflection subtraction based on two successive images or using the initial image as a reference, were compared (triangles vs continuous lines) with no apparent difference. Finally, the two devices (spectrometer and camera) were also compared, providing the same results (crosses for the spectrometer, lines and triangles for the camera).

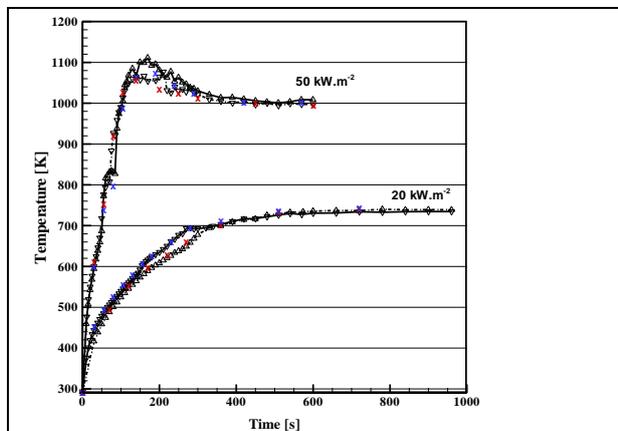


Figure 8. Time evolution of the surface temperature for two incident fluxes (20 and 50 kW/m²).

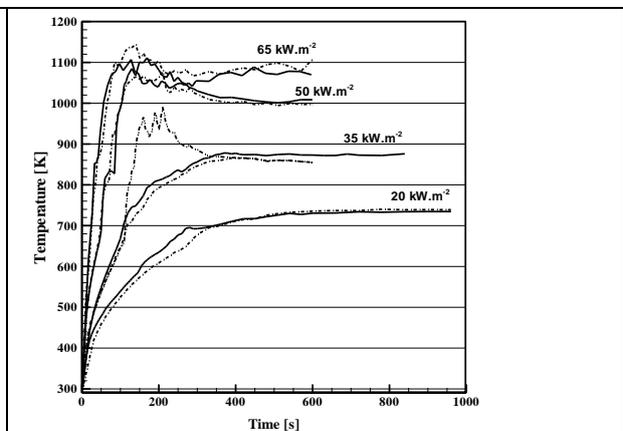


Figure 9. Time evolution of the surface temperature for four incident fluxes: 20, 35, 50 and 65 kW/m²

Continuous lines and triangles for the camera data, crosses for the spectrometer data. Two sets of results for each flux (repeatability test).	(camera data). One particular curve at 35kW/m ² was obtained using piloted ignition after 100s of degradation (shifted from the other curves between 100 and 300s because of the flame contribution).
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These tests reported in Figure 8 give some confidence for the whole experimental campaign. Then, Figure 9 allows comparing the sample evolution as a function of the incident flux, still presenting two tests in each case with the same flux conditions. The curves show that the increase in temperature was particularly sharp and a plateau was always reached between 300s and 400s as a consequence of the energy balance. Some fluctuations appeared for the highest fluxes after 100s as a consequence of ignition at the sample surface, which provided a supplementary incident flux for the degradation. It did not happen in standard conditions below 35 kW/m², but it was forced using a piloted ignition device in one special test (curve shifted from the two others at 35 kW/m²). This showed the influence of the flame developing along the sample. When the flame disappeared after 300s, the curve tended toward the same plateau than the two others. The temperature increase was unexpectedly sharp: several hundreds of degrees in the first minute, further increasing with the incident flux. This was confirmed by both apparatuses with independent measurements.

These data will be useful for the model validation, but the sharp increase during the first step shows that further experiments should be conducted decreasing the acquisition time, which is planned for the next experiments.

Conclusion

The surface temperature of a carbon composite sample was identified during its degradation under a cone calorimeter, based on infrared observation with an IR camera and a FTIR spectrometer simultaneously. A special subtraction process was applied on spectra and images in order to eliminate the parasitic signal due to the reflection of the incident radiation by the sample. This non intrusive method gave the time varying evolution of the temperature during a series of degradation experiments for incident fluxes between 20 and 65 kW/m². Both apparatuses independently provided results showing a sharp temperature increase when the sample is irradiated. The present method gave promising results, which should be now confirmed and refined with a shorter time step acquisition in further experiments.

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Acknowledgements:

The research leading to these results has received funding from the European Union's Seventh Framework Programme (FP7/2007-2013) for the Fuel Cells and Hydrogen Joint Technology Initiative under grant agreement n° 325329.