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Directional spectral reflectivity measurements of a carbon fibre reinforced composite up to 450 °C

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ABSTRACT

A quantitative study of the bi-directional reflectivity of a carbon/poly-ether-ether-ketone (PEEK) based composite is performed at room temperature using a Fourier Transform Infra-Red Bruker 80v spectrometer (0.6–25 μm) angles of incidence and collection ranging from 11 to 83° and by varying fibre orientation. Then, a home-made compact cell, based on a customized resistive heater, is adapted to the sample compartment of the spectrometer for measuring the temperature dependency of the normal reflectivity of the composite sample from 20 to 450 °C. A complete validation of the thermal performances of the cell is thus presented to support the interpretation of the previous reflectivity measurements. The respective contributions of the carbon fibrous reinforcement and of the PEEK matrix on the reflective behaviour up to 450 °C is finally discussed. Temperature is shown to play a minor role on the optical properties of the tested materials, thus indicating a predominant role of the carbon fibres. Conversely, the room temperature angular study shows the high importance of the beam incident angle with respect to fibres orientation and enables to characterize this strongly anisotropic reflective behaviour.

1. Introduction

Near infrared laser diodes are promising heating methods to replace traditional gas torches in Automated Fibre Placement (AFP) machines for manufacturing thermoplastic carbon fibre composites (TP-CRFP) [1]. Indeed, thanks to their ability to deliver uniform high fluxes over large surfaces, they can provide effectively the high energy required to achieve the fusion bonding of the composite tapes [2]. In order to optimize the energy efficiency of the laser assisted processes such as AFP, the thermal radiative properties of the material involved must be known according to the laser incidence, its wavelength, the fibre orientation and the material temperature. Yet, whereas the mechanical and chemical properties of (TP-CRFP) have been thoroughly studied in literature [3], the radiative aspects concerning such materials remains poorly documented.

Previous studies have highlighted the strong back-scattering power of CRFP due to the presence of cylindrical fibres [4]. Hence it has also been shown that simple specular reflection assumptions fail to correctly model the surface/laser interaction [5,6]. The diffusive behaviour is influenced by the angle of incidence of the beam Θ. To illustrate this behaviour, directional hemispherical reflectivity measurements have been performed at room temperature with the beam aligned to the fibre directions at 0.5, 1, 1.5 and 2 μm on a carbon/PEEK composite by varying the polar angle Θ between 10 and 80° [7]. It was found that the reflected flux rises from 0.1 to 0.6 when the beam is shifted from normal incidence to a grazing one. Moreover, similarly to the pioneering experiment carried out by Grouve [6], the same authors qualitatively observed, for an incidence of 65°, that the scattering profile of the composite surface is highly dependent of the angle, ϕ, between the axis of the fibre and the axis of the laser. The role of the polymer rich surface was also studied in Hohmann et al. [4]. Total directional reflections measurements were performed at 500 nm with integrating spheres on carbon fibre laminates, one where the polymer matrix at the surface was removed by laser processing, and the other one with the polymer layer at the surface. Results showed that the normal total reflectivity was 0.02 higher for the latter sample. The authors explained this reflectivity increasing by the fact that the matrix alone has a higher refractive index so that a little less light is absorbed. Eventually, in parallel to experimental studies on composite materials, optical models based on Ray Tracing [7] or Maxwell Equations [4] were developed. Nevertheless, whatever

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the primary assumptions, they all required the accurate knowledge of the optical indexes n and k of the material that can only be obtained with fine optical measurements.

From this point, it appears that still few studies dealing with the high temperature radiative properties (up to 450 °C) of CFRP have been carried out. Nevertheless, it is expected the radiative properties of CFRP can evolve, especially when the PEEK matrix melts (around 343 °C) or due to thermal degradations at higher temperatures [8,9].

The knowledge of the effects played by the temperature on the radiative properties are therefore crucial for bringing accurate data in order to run thermal modelling works. One can note the preliminary work of Grouve [6], who used a crude resistive heater placed under a carbon/(Polyphenylene sulphide) PPS composite sample in order to perform hemispherical reflectivity measurements for an incidence of 66° with respect to the normal axis of the sample surface and at 1080 nm only, between 25 and 350 °C. The author qualitatively observed negligible variations but little information was given on the performances and thermal homogeneity of the heating apparatus. On the other hand, limiting the campaign of radiative measurements only at the wavelength of the impinging laser reduces the exactness of the thermal modelling since hot CFRP tapes are also likely to emit thermal radiation in the infrared spectral range where Plank's law predicts significant emission around 450 °C.

Many examples of reliable heating systems developed to perform high temperature (up to 2500 °C) emissivity measurements by spectroscopic method can be found in the literature for materials either semi-transparent or opaque [10–18]. In the following part, the focus is put on the experimental developments concerning opaque materials. In particular, the need to measure the radiative heat flux emitted by a sample and a blackbody in the exact same thermal conditions has prompted the authors to pay a special care to the thermal homogeneity delivered by their heating systems. For opaque samples in thermodynamic equilibrium, the reflectivity is deduced from a direct emissivity measurement with the Kirchhoff's law of thermal radiation. For instance, in [11], Del Campo et al. reported a thermal stability of less than 2 °C at 807 °C. For spectral measurements, the experimental set-up are generally built around a FTIR spectrometer, which is equipped with a set of high sensitivity detectors in order to cover a wide spectral range. The choice of the heating method depends on the temperature range of interest. Hence, various techniques can be encountered such as small furnaces [11,12] for moderate temperature (from 20 up to 1000 °C) [19] and laser heating for reaching higher temperature range (up to 2000 °C) [10,12,19]. Directional analysis can also be performed through rotating sample holders placed in the measurement chamber [11,12]. For reflectivity measurements below 1000 °C [19], the set of light sources inserted within the spectrometer can also be used to illuminate successively the sample and the reference mirror. In [20], the authors measured the reflectivity of semi-transparent materials in the far-infrared range up to 500 °C with a FTIR spectrometer equipped with a thin ceramic plate, heated through an electrical resistance inserted in its middle part, and mounted on the sample holder. Such approach worked also well in [21] where a commercial resistive heater is used as a sample holder to study the optical properties of thermochromic samples up to 150 °C. Nevertheless, in both cases, no clear direct thermal validation of the set up performances is given. In the following, the principle remains of particular interest because it takes advantage of the high sensitivity of the FTIR spectrometer and scans the temperature range of interest.

In order to refine the analysis of the reflective behaviour of a carbon/PEEK composite, the following study aims firstly at quantifying the reflected intensities along the diffusion profile of an incident beam, for various tape orientation, through a FTIR spectrometer equipped with a directional measurement unit. The spectral range covered by the spectrometer goes from 0.66 up to 25 μm. The influence of the fibre orientation is analysed for incident angles ranging from 15 to 83 °C. Secondly, an original compact cell, based on a customized resistive heater, is proposed in this work to investigate the evolution of the normal reflectivity with temperature. The cell is inserted in the sample compartment of the available FTIR spectrometer and allows performing measurements on opaque materials from 20 up to 600 °C. After the determination of the thermal and optical performances of the developed heating unit, the results obtained on the carbon/PEEK composite are presented. Finally the role of each phase of the composite sample on its global radiative behaviour will be discussed.

2. Experimental procedure

2.1. Description of the infrared spectrometer

Before describing the experimental set-up used to perform the measurements, let us define the main radiative quantities of interest in this study. Fig. 1 shows how the incident beam can interact with the surface of the composite. With respect to the normal axis of the plane bearing the CFRP sample, let us define θ_i the polar incident angle and the azimuthal angle ϕ_i, which is the deviation angle with the main direction of the unidirectional fibres. The bi-directional reflectivity function BRDF, $f_\lambda(\theta_r, \phi_r, \theta_i, \phi_i, T)$, is defined by:

$$f_\lambda(\theta_r, \phi_r, \theta_i, \phi_i, T) = \frac{I_{\lambda,r}(\theta_r, \phi_r, \theta_i, \phi_i, T)}{I_\lambda(\theta_i, \phi_i, T)\cos\theta_i d\Omega_i} \quad (1)$$

where $I_{\lambda,r}(\theta_r, \phi_r, \theta_i, \phi_i, T)$ is the reflected spectral intensity in direction (θ_r, ϕ_r) due to the irradiance $I_\lambda(\theta_i, \phi_i, T)\cos\theta_i d\Omega_i$ in the incident direction (θ_i, ϕ_i). Here T stands for the temperature and $d\Omega_i$ is the elementary solid angle around direction (θ_i, ϕ_i). The spectral directional hemispherical reflectivity, $\hat{\rho}_\lambda(\theta_i, \phi_i, T)$ is given by:

$$\hat{\rho}_\lambda(\theta_i, \phi_i, T) = \int_{2\pi} f_\lambda(\theta_r, \phi_r, \theta_i, \phi_i, T)\cos\theta_r d\Omega_r \quad (2)$$

where $d\Omega_r$ is the solid angle around (θ_r, ϕ_r).

When the optical response of a material is purely specular, all the reflected flux is located on the *zygomorphic* axis, *i.e.* on the axis symmetric to the incident axis with respect to the normal axis, and:

$$\phi_r = \phi_i \quad (3)$$

The optical measurements were performed with a Fourier Transform Bruker 80v infrared spectrometer. Fig. 1 represents the optical paths allowed by the three measurements units available:

- Unit U_{NN} gives access to normal - normal specular reflectivity measurements ($\rho_{\lambda,NN}$)[22] given by Eq. (2) when
- $\theta_i = \theta_r = 0°$. It must be mentioned that, in this unit, the beam is not strictly normal to the surface but slightly shifted (11°) in reason of the optical configuration.
- Units U_{NH} are Teflon and gold coated integration spheres, which allow respectively the measurements of the near and mid infrared normal - hemispherical reflectivity $\rho_{\lambda,NH}$ given by Eq. (2) when $\theta_i = 0°$.
- Unit U_{BDR}: automated variable angle reflection accessory, which permits to measure the bi-directional reflectivity $\rho_\lambda(\theta_r, \phi_r, \theta_i, \phi_i, T)$ for $\phi_r = \phi_i$ and, when the geometry of illumination is determined [23], to characterize bi-directional reflectivity functions (BDRF) for variable couple of incident and reflected angles (13–85°).