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A framework for evaluating the thermal behaviour of carbon fibre composite materials

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Abstract

This paper presents a framework for evaluating the thermal behaviour of carbon fibre composite materials, based on a model of minimum complexity able to capture the thermal evolution of the particular material, and estimate rates of mass loss due to pyrolysis. An experimental programme consisting of Cone Calorimeter tests is used to evaluate the prediction capabilities of the model. This approach is well suited to practical applications in the design and assessment of the performance of complex composite fibre/polymer reinforced materials, thereby helping to ensure sufficient fire safety performance.

Introduction

Carbon fibre composite materials are commonly used in high pressure cylinders for storage and transport of hydrogen. Despite significant strength, low burst potential and low weight of these vessels, they may be vulnerable to fire due to the combustible nature of the constituent materials. Understanding the performance of the composite material in fire is essential to prevent bursting of the vessels and the associated consequences. Furthermore, this will allow development of new fire protection strategies and improvement of current standards. This work is specifically focussed on developing the tools that will allow the prediction of the thermal behaviour of hydrogen pressure vessels with stationary and transport application during the design stage considering an arbitrary temporally and spatially varying fire boundary condition.



Fig. 1. Vessel of carbon fibre composite during manufacturing

It has been demonstrated that existing models of thermal decomposition can introduce large uncertainties due to the number of parameters required to characterise the material and combustion environment (i.e. material parameters which determine these phenomena) [1,2]. Therefore, this work explores a novel simplified method to analyse the coupled mechanisms of heat transfer and thermal degradation for carbon fibre composite materials under controlled conditions of heat exposure. The application of this methodology is based on the combination of small scale Cone Calorimeter and thermogravimetric experiments, through a two-step uncoupled analysis in order to quantify the rates of mass loss/pyrolysis.

A model of minimum complexity is presented, which captures the key physical processes required to obtain

agreement with the experimental temperature data. This is assessed using measurements from the Cone Calorimeter to determine the suitability in recreating the in-depth temperature measurements and the mass loss rate.

Experimental set-up and main outcomes

A primary experimental programme based on the use of the Cone Calorimeter [3] was performed so as to provide (1) quantification of flammability parameters, (2) a baseline estimation of the thermal properties, and (3) an assessment of the combustibility of the carbon fibre composite. Samples of thickness 4.5 and 29 mm were tested using the standard sample holder with aluminium foil and two different materials at the rear surface: (1) a 25 mm thick ceramic insulation and (2) a 25 mm thick aluminium block. The dimensions of the samples were 10 cm by 10 cm. Experiments were run up to external radiant heat fluxes of $80 \text{ kW}\cdot\text{m}^{-2}$, and using a pilot spark to ignite the flammable mixture formed at the surface. An illustration of a sample prepared for this programme and its residue after flame out is shown in Fig. 2. An extensive analysis of the results is not presented here; nevertheless, a qualitative description of the material behaviour will be presented at the end of this section.

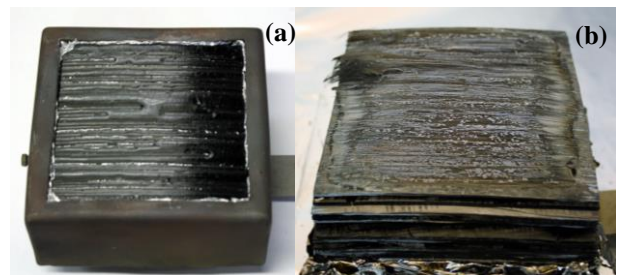


Fig. 2. 29 mm thick sample of carbon fibre composite for high pressure cylinders (a) before, and (b) after, a piloted flammability experiment at $30 \text{ kW}\cdot\text{m}^{-2}$

The experimental programme using the Cone Calorimeter was extended in order to characterise the thermal evolution of the carbon fibre composite samples. Further instrumentation such as K-type and N-type thermocouples were inserted at different thicknesses. An

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aluminium block was used so as to provide a better characterisation of the rear surface boundary condition [4], and ceramic insulation paper was used as lateral boundary element instead of the standard sample holder. Experiments were run at external incident heat fluxes of $10 \text{ kW}\cdot\text{m}^{-2}$ and $30 \text{ kW}\cdot\text{m}^{-2}$ so as to present case studies without and with thermal degradation, respectively. Larger heat fluxes were not explored, and the pilot spark was not used, since no flaming was pursued so that the level of uncertainty with regard to the boundary condition definition could significantly be reduced. Three to four repetitions were performed in order to assess the consistency of the obtained results. Experiments with no thermocouples were carried out to obtain precise mass loss measurements. The sample preparation and experimental set-up are presented in Fig. 3. Detailed results will be discussed in further sections.

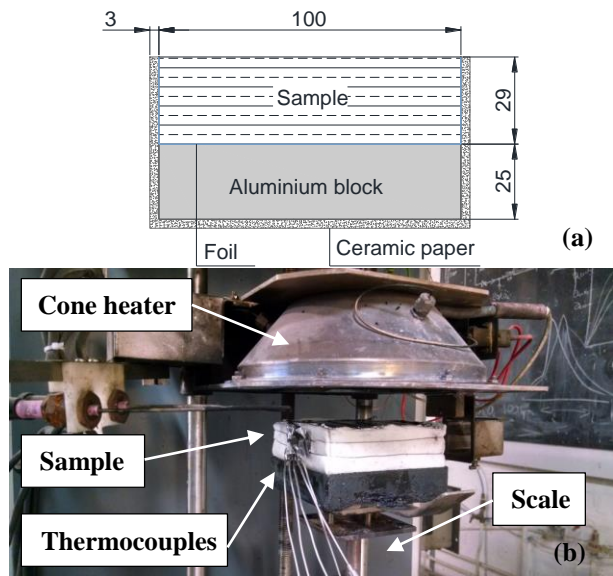


Fig. 3. (a) Sample configuration – units in mm (b) Experimental set-up in the Cone Calorimeter using thermocouples

A summary of the observed material behaviour based on both experimental programmes is presented below:

- The epoxy experiences pyrolysis, with a residue remaining, which consists of mainly carbon fibres and presumably char from the epoxy. A residue from a test at $30 \text{ kW}\cdot\text{m}^{-2}$ is shown in Fig. 2.
- The rate of pyrolysis is characteristic of a thick charring material, with an initial peak followed by a decay [5,6].
- The volume from the sample barely modifies after pyrolysis and flame out, with a slight increase (approximately 1 to 2 mm), presumably due to the delamination of the carbon fibre layers.
- Oxidation rates, understood as the regression of the surface residue, are shown to be negligible for the studied conditions.
- Temperature measurements within the sample show no smouldering processes within the material, therefore permeability seems low.

- No endothermic plateaus are observed in the temperature measurements within the sample for the studied conditions of heat exposure.

Simplified thermal model

A simplified model is proposed for predicting the thermal behaviour and pyrolysis rates of the particular studied material. The approach is based on isolating the heat transfer problem and the chemistry of the pyrolysis reactions.

Hypotheses

The proposed methodology applies to a series of hypotheses that shall be met by the behaviour of the material, such as:

- (1) Opaque material, thus no transmission of radiation through thickness,
- (2) negligible change in volume after pyrolysis,
- (3) insignificant oxidation rate for a wide range of temperatures or non-oxidative conditions,
- (4) thermal profile without endothermic plateaus and
- (5) low permeability, thus negligible convective heat transfer through thickness.

The energy balance – inert behaviour

The governing equation for the one-dimensional definition of the studied problem corresponds to the heat diffusion equation presented below:

$$\frac{\partial k \partial T}{\partial x^2} = \rho c \frac{dT}{dt} + \dot{g}''' \quad (1)$$

where k , ρ and c are the thermal conductivity, density and heat capacity of the material, and \dot{g}''' is the volumetric term of energy generation/consumption. If the aforementioned hypotheses are met, and the conductivity is considered constant, Eq. (1) can be represented as the energy balance defined in Eq. (2) for discretised elements, which is schematically illustrated in Fig. 4.

$$\dot{q}''_{cond_{i-1 \rightarrow i}} = \dot{q}''_{cond_{i \rightarrow i+1}} + \dot{q}''_{st_i} + \Delta x \cdot \gamma \cdot \rho_0 \cdot \frac{\dot{\omega}_{p,i}}{\Delta H_p} \quad (2)$$

where Δx is the element thickness, $\dot{q}''_{cond_{i-1 \rightarrow i}}$ and $\dot{q}''_{cond_{i \rightarrow i+1}}$ are the heat conducted from the discrete element $i-1$ to i , and from the discrete element i to $i+1$, \dot{q}''_{st_i} is the heat stored in the discrete element i , γ is the fraction of material that can be pyrolysed, ρ_0 is the virgin density of the material, $\dot{\omega}_{p,i}$ is the pyrolysis rate from the element i , and ΔH_p is the heat of gasification for the material that can be pyrolysed.

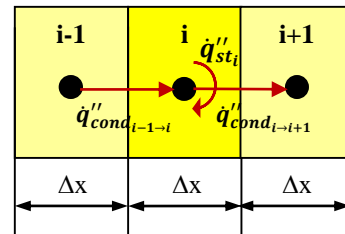


Fig. 4. Energy balance in the finite difference domain

The pyrolysis chemistry is often considered by the community as an Arrhenius expression for each reaction step:

$$\dot{\omega}_{p,i} = A \cdot \exp(-E_a/RT) \cdot f(\alpha) \quad (3)$$

where A is the pre-exponential factor, E_a is the activation energy, R is the ideal gas constant, T is the temperature,

and $f(\alpha)$ is the conversion function, which is dependent on the kinetic mechanisms of the reaction [7]. Nonetheless, the approach proposed by the authors is based on considering that the material behaves inertly, thus the following expression being applicable:

$$\frac{\partial k \partial T}{\partial x^2} = \rho c_{eff} \frac{dT}{dt} \quad (4)$$

where c_{eff} is the effective heat capacity of the material, which considers the energy required to increase the temperature of the material, and overcome the endothermicity from the pyrolysis reaction. The form of the effective heat capacity can be considered as a temperature dependent function.

Eventually, the discretised version of Eq. (4) for a temperature dependent thermal conductivity is obtained as:

$$k(T_i) \cdot \frac{T_{i-1}^j - 2 \cdot T_i^j + T_{i+1}^j}{\Delta x^2} + \frac{\delta k(T_i^j)}{\delta T} \cdot \left(\frac{T_{i+1}^j - T_{i-1}^j}{2 \cdot \Delta x} \right)^2 \quad (5)$$

$$= \rho(T_i^j) \cdot c_{eff}(T_i^j) \cdot \left. \frac{dT}{dt} \right|_i$$

where T_{i-1}^j , T_i^j and T_{i+1}^j are the temperature of the element $i-1$, i and $i+1$ respectively for the time step j . A tridiagonal system may be obtained by applying the Crank-Nicolson method, which simplifies the problem solution, and allows for an optimisation of computational costs.

Inverse modelling effective thermal properties

The heat transfer problem, defined by Eq. (5), requires a series of material properties such as thermal conductivity, density and effective specific heat capacity. While the density is a parameter that can be easily measured, quantification of the conductivity and specific heat capacity is often tedious due to the need of particular apparatuses [8,9], and post-processing techniques, so as to validate the applicability of these parameters.

Inverse modelling may offer a practical solution for case studies where these parameters cannot be measured directly. Nevertheless, *Bal et al.* [1] recently showed that the use of inverse modelling techniques introduces compensation errors in an analysis for determining parameters of a thermal model such as *Gpyro* [2], which are due to the large number of unknown parameters. These compensation errors are clearly conditioned by an uncertain definition of the boundary condition. For the particular case that considers an effective irradiation \dot{q}_e'' , the boundary condition can normally be approximated as:

$$\alpha \cdot \dot{q}_e'' + h_c \cdot (T_\infty - T_s) - \varepsilon \cdot \sigma \cdot (T_s^4 - T_\infty^4) \quad (6)$$

$$= -k \cdot \left. \frac{\delta T}{\delta x} \right|_{x=0^+}$$

where α is the absorptivity, ε is the emissivity, h_c is the convective heat transfer coefficient, T_∞ and T_s is the ambient and surface temperature, respectively, and $\left. \frac{\delta T}{\delta x} \right|_{x=0^+}$ is the thermal gradient at the surface.

When applying inverse modelling techniques so as to fit the experimental time-history of temperatures within the sample, defined as the particular problem presented as Eqs. (5) and (6), the algorithm tends to converge to a local minimum which provides a confident thermal diffusivity. If the boundary condition is not accurately defined, a wrong value of thermal conductivity may be

obtained so as to provide a consistent energy balance at the surface. As a result, the volumetric heat capacity will be altered to condition a thermal diffusivity that transfers the heat as the characteristic experimental thermal wave. Therefore, even when the number of parameters in the inverse modelling problem is reduced, non-realistic solutions can be obtained, which may provide a good fitting of the experimental results.

Thus, the use of inverse modelling techniques is only ideal if either thermal conductivity or volumetric heat capacity are known. Values of thermal conductivity obtained using a *Holometrix TCHM-LT* apparatus [10] are used as a reference to assess the consistency of the inverse modelling results. For simplicity, the *Levenberg-Marquardt algorithm* (LMA) [11,12] is used for the inverse calculation of the effective thermal properties of the material.

Methodology application

The proposed methodology considers a two-step uncoupled analysis, by which the heat transfer problem is solved in the first instance, followed by the estimation of the fraction of remaining mass and pyrolysis rates. The fraction of remaining mass is defined as a function of time and thickness.

The studied material is considered as a series of finite difference elements, which have a known bulk temperature once the heat transfer is solved. Each element is assigned a value of mass as a function of its temperature, therefore defining a time-history of mass loss. Integrating the mass over the thickness of the sample for each time step, the overall evolution of mass from the sample can be obtained as shown in Eq. (7):

$$m^j = \frac{\sum_{i=1}^N (m_i^j \cdot \Delta x_i)}{L} \quad (7)$$

where m^j is the mass of the sample at the time step j , m_i^j is fraction of remaining mass for the discrete element i at the time step j , Δx_i is the thickness of the discrete element i , N is the total number of discrete elements, L is the sample thickness. Applying differentiation over time, the mass loss rate or rate of pyrolysis can be obtained.

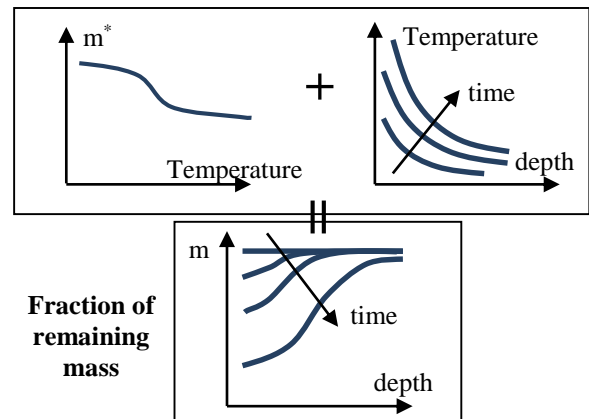


Fig. 5. Conceptual definition for simplified thermal model, based on TGA analyses and measurement of thermal profile

Whereas Arrhenius expressions could be used to determine the rate of mass loss for each element as a function of its temperature time-history, for simplicity

the approach proposed for the present work is to consider the TGA data as a reference for assigning the fraction of remaining mass. The conceptual definition of this approach is presented in Fig. 5.

Uncertainties

A series of uncertainties are encountered when applying this methodology; (a) with regard to the data obtained from experimentation:

- *Reliability in the thermocouples positioning.* Inaccurate positioning may lead to incorrect values of thermal properties, especially with thin samples. A precise drilling system is used in order to reduce the uncertainty of the positioning to ± 2 mm. Additionally, samples are chopped after the experiment in order to verify the actual thermocouple location.
- *Resolution of temperature measurements through thickness.* A sparse density of monitoring positions may lead to incorrect values of thermal properties. Generally, six temperature measurements per sample are used, with higher density near the surface where higher thermal gradients are expected. Additionally, results from repeated experiments are combined to assess the consistency of the thermal properties.
- *Boundary condition.* The main uncertainty in the definition of the boundary condition lies in the quantification of the convective heat transfer coefficient, since values of emissivity/absorptivity are provided by CNRS/LEMETA, and the external heat flux is measured with an expanded uncertainty of $\pm 3\%$. The convective coefficient is obtained considering a Nusselt correlation for the horizontal hot plate [13,14]. An uncertainty of $\pm 20\%$ is used to evaluate the consistency of inversely calculated thermal properties with thermal conductivity measurements and the thermal inertia assessment from flammability experiments.

(b) With regard to the material:

- *Homogeneity of the fraction of epoxy within the sample.* Manufacturers may be able to provide an assessment of this homogeneity. However, if this information is not available, it can be assessed by analysing the consistency between experimental repeats.
- *Fraction of epoxy for the composite material.* Since the pyrolysis is essentially related to the epoxy, an inaccurate fraction of epoxy may lead to wrong estimations of pyrolysis rates. This is assessed by considering an uncertainty region in the fraction of epoxy given by TGA analyses.

(c) With regard to the simplified thermal model:

- *Validity of effective thermal properties for other regimes of heat transfer.* The validity of the effective thermal properties is restricted up to the conditions tested.
- *Suitability of the TGA data, in terms of the heating rate.* An assessment of the expected

heating rates shall be performed so as to validate the methodology approach. The suitability of an uncertainty region based on TGA results with different heating rates will be discussed.

- *Oxidative conditions.* If a significant oxidative behaviour is obtained in the solid-phase this approach is expected to fail in providing accurate predictions.

Results and discussion

First, the heat transfer modelling based on the inverse calculation of effective thermal properties is discussed. Thereafter, the prediction capability of the uncoupled pyrolysis model is assessed.

Step 1: Heat transfer modelling

Fig. 6 shows the experimental (dashed lines) and modelled (solid lines) time-history of temperatures within the material for an external heat flux of $10 \text{ kW}\cdot\text{m}^{-2}$. The shading indicates the modelled temperatures within a deviation of ± 1 mm from the nominal depth. These conditions of heat exposure represent a case study where no thermal degradation is obtained. Therefore, the approach is based on considering constant effective thermal properties. Good agreement is observed between the experimental measurements and the model, indicating the general rationality of an approach based on constant thermal properties for these conditions.

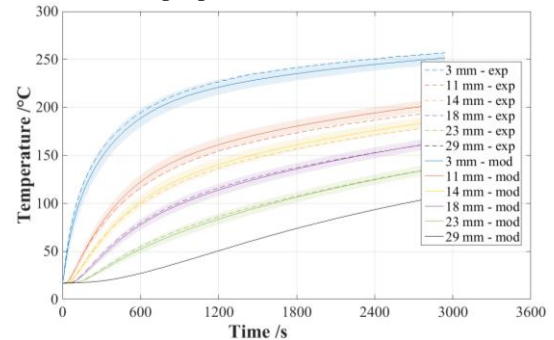


Fig. 6. Experimental and modelled temperatures at $10 \text{ kW}\cdot\text{m}^{-2}$

Fig. 7 shows the experimental (dashed lines) and modelled (solid lines) time-history of temperature measurements within the sample for an external radiant heat flux of $30 \text{ kW}\cdot\text{m}^{-2}$. This heat exposure represents a case study where thermal degradation is obtained. Therefore, the approach based on constant effective thermal properties is expected to fail in providing an accurate prediction of the thermal gradients through thickness. An approach based on a constant thermal conductivity and a temperature dependent heat capacity as shown in Eq. (9) is used:

$$k(T) = k_0 \quad (8)$$

$$c_{eff}(T) = c_0 \cdot \left(\frac{T}{T_r}\right)^{c_n} \quad (9)$$

where c_0 is the specific heat capacity at the reference temperature T_r , and c_n is the index that determines the growth or decay of the specific heat capacity. For simplicity, a constant thermal conductivity is proposed so as to reduce the degrees of freedom in the inverse modelling. Moreover, a temperature dependent thermal

conductivity failed in providing rational trends between the model and the experimental results.

Generally, a good agreement is again observed between the experimental results and the model. Slight discrepancies in the trend are observed for inner positions at earlier stages of the experiment (<300s). The experimental temperature near the surface indicates a clear change in trend after 1200 s, likely due to the detachment of different layers in the composite material (delamination), thus resulting in a slight increase of volume. This phenomenon is also observed for the positioning at 4 mm between 450 and 750 s.

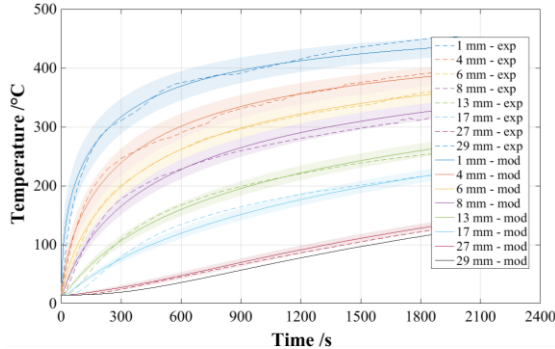


Fig. 7. Experimental and modelled temperatures at 30 kW·m⁻²

Table 1 shows the range of effective thermal properties obtained from different techniques. When attempting to obtain constant thermal properties, a good agreement is observed between the thermal inertia analysis and inverse modelling at 10 kW·m⁻². Two approaches are considered for the latter: (1) assuming values of conductivity and optimising the convective coefficient and specific heat capacity, and (2) assuming a convective coefficient as a function of the Nusselt number with an uncertainty region and optimising conductivity and heat capacity. Low values of conductivity are obtained for the higher boundary of the convective coefficient, suggesting an overestimation of heat losses. An increasing heat capacity is obtained for 30 kW·m⁻², making evident the endothermicity of the pyrolysis; whereas the optimised constant thermal conductivity is in the measured range observed for lower temperatures.

Table 1. Effective material properties

Method/ Property	Ignition theory	Inverse modelling		
		10	10	30
Heat flux /kW·m ⁻²	~	10	10	30
h_c /W·m ⁻² ·K ⁻¹	f(Nu)	8±3 (optim.)	f(Nu)	f(Nu) ±20%
k /W·m ⁻¹ ·K ⁻¹	0.41-0.61 (measured in range 51-174°C)	0.41-0.61 (assumed)	0.19-0.52 (optim.)	0.48 ±0.03 (optim.)
c /J·kg ⁻¹ ·K ⁻¹	700-1300 (calc.)	1001±99 (optim.)	871 ±187 (optim.)	389(±28)· (T/20) ^{0.76(±0.06)} (optim.)
k_{pc} /W ² ·s·K ⁻² ·m ⁻⁴	6.59·10 ⁵ ±0.73·10 ⁵ (optim.)	7.01·10 ⁵ ±1.79·10 ⁵ (calc.)	4.25·10 ⁵ ±2.04·10 ⁵ (calc.)	~

Step 2: Mass loss rate modelling

The obtained effective thermal properties are used to solve the heat transfer problem directly, and then determine the rate of mass loss for the 30 kW·m⁻² case study. Fig. 8 shows the rates of mass loss for four repeated experiments. Good consistency is observed between the experiments; however, it should be noted that their durations are different. This is due to the sudden auto-ignition of the mixture at the surface of the material, thus only data prior to ignition being presented. The modelled mass loss rate is presented as an uncertainty region delimited by the model results using TGA data in nitrogen and at heating rates of 2.5 and 20°C·min⁻¹.

In general, a good agreement is observed between the experimental and modelled mass loss rate. An underestimation or delay is observed for early stages of heat exposure (<120 s), which could be attributed to a bad prediction of the thermal gradients near the surface during the early stages of the test, or a careless non-dimensionalisation of the TGA data. Indeed, the model using TGA data at a significantly low heating rate, e.g. 2.5°C·min⁻¹, is intuitively likely to overestimate the rate of mass loss and not underestimate it.

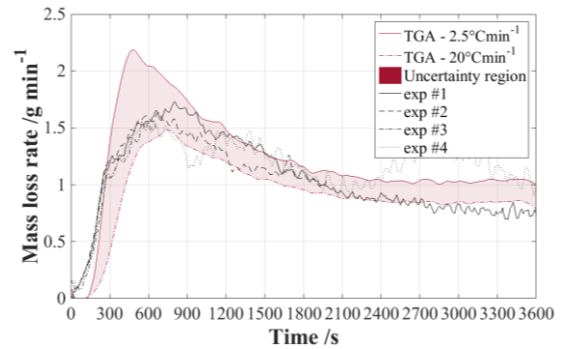


Fig. 8. Experimental and modelled mass loss rate at 30 kW·m⁻²

Fig. 9 shows the obtained rates of temperature variation at different depths. It is shown that heating rates over 20°C·min⁻¹ are only observed for very early stages of the heat exposure (<200 s), and mainly for the first 5 mm of material. Therefore, the simplistic approach considering a direct relationship between mass and temperature seems to be effective for materials of these characteristics. This characteristic behaviour is justified by the insignificant change in volume after pyrolysis, thus reducing the net heat through thickness due to the fast increase of temperature at the exposed surface.

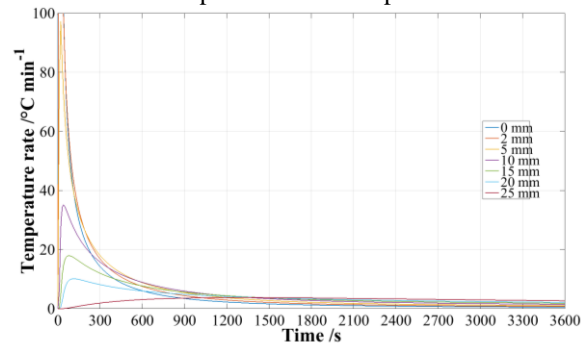


Fig. 9. Heating rate for different thicknesses within a sample exposed to 30 kW·m⁻²

The rates of mass loss shown in Fig. 8 were obtained considering a fraction of remaining mass in the composite of 70%. However, TGA results often show a fraction varying from 60% to 80%. Fig. 10 shows the modelled rates of mass loss considering different fractions. It is observed that a deviation of $\pm 10\%$ defines a significant uncertainty region. Therefore, a precise definition of this parameter seems crucial. Results presented in Fig. 8 indicate that a 70% ratio seems to be a reasonable average value for this material.

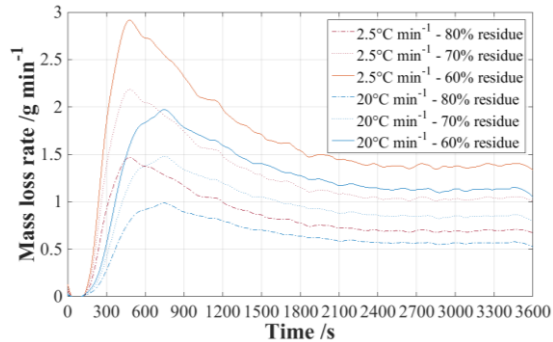


Fig. 10. Modelled mass loss rates considering different fraction of residue and TGA curves

Conclusions and further work

A simple model for the determination of the thermal behaviour of a carbon fibre composite material has been presented. The methodology is based on a two-step uncoupled analysis that first solves the heat transfer considering the material inert, and later on assigns a specific fraction of remaining mass as a function of the temperature.

Effective thermal properties, optimised using inverse modelling techniques, are shown to provide a reasonable prediction of the thermal evolution of the material. Good predictability of the rates of pyrolysis is provided by the uncoupled analysis using TGA data. The effectiveness of the approach is justified by the negligible change in volume following pyrolysis. This causes the heating rate through thickness to fall within the range normally used for thermogravimetric experiments. However, it is found that the uncertainty in the percentage of residue is shown to have a significant effect.

This methodology presents a potential tool for evaluating rates of pyrolysis of materials with similar material characteristics, or charring materials under non-oxidative conditions. The applicability of this method is apparent for large-scale experiments in which only measurements of the temperature in the solid-phase are required; thus, not having to solve the complete heat transfer problem which the global uncertainty is very high due to the extended uncertainty in accurately defining the boundary condition.

Further work is required in order to assess the suitability of the presented methodology for more severe conditions of heat exposure, thus higher heating rates through thickness. This would be vital in order to find a generic function of effective thermal properties for any arbitrary heating scenario, and to validate the uncoupled simplified pyrolysis model.

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