Pyrolysis and spontaneous ignition of wood under transient irradiation: Experiments and a-priori predictions

Izabella Vermesi\textsuperscript{a}, Matthew J. DiDomizio\textsuperscript{b}, Franz Richter\textsuperscript{a}, Elizabeth J. Weckman\textsuperscript{b}, Guillermo Rein\textsuperscript{a,∗}

\textsuperscript{a} Department of Mechanical Engineering, Imperial College London, SW7 2AZ London UK
\textsuperscript{b} Department of Mechanical and Mechatronics Engineering, University of Waterloo, Waterloo, ON, Canada N2L 3G1

\section*{A R T I C L E   I N F O}

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\section*{A B S T R A C T}

Wood is a material widely used in the built environment, but its flammability and response to fire are a disadvantage. Therefore, it is essential to have substantial knowledge of the behavior of wood undergoing external heating such as in a fire. The majority of studies in the literature use constant irradiation. Although this assumption simplifies both modelling and experimental endeavors, it is important to assess the behavior of materials under more comprehensive heating scenarios which might challenge the validity of solid-phase ignition criteria developed previously. These criteria are evaluated here for the spontaneous ignition under transient irradiation by combining experimental measurements and a-priori predictions from a model of heat transfer and pyrolysis. We have applied a two-step transient irradiation in the cone calorimeter in the form of a growth curve followed by constant irradiation. We use white spruce samples of size 100×100 mm and thickness of 38 mm. We measure the temperature at different depths and the mass loss. A one dimensional model written in the open source code Gpyro is used to predict the pyrolysis behavior. The model has a chemical scheme in which the components of wood (hemicellulose, cellulose, lignin) become active, then decompose in two competing reactions: one reaction to char and gas, and one reaction to tar. The kinetic parameters, as well as the thermal properties of the wood and char are taken from the literature, while ρ and moisture content are measured experimentally. A priori predictions of the temperature, made prior to the experiments, show excellent agreement with the measurements, being within the experimental uncertainty range. The mass loss rate (MLR) predictions are qualitatively similar to the measurements, but there is a large uncertainty in the measurements. For a-posteriori simulations, certain parameters are changed after having access to the measurements to improve the simulations. Also, we perform an evaluation of the solid phase ignition criteria used in the literature, and find that neither criteria is a consistent indicator of ignition. These results help understand the spontaneous ignition of wood subjected to transient irradiation and identify strengths and gaps in the topic.

\section*{1. Introduction}

Wood is one of the oldest building materials and, because of its cost effectiveness and environmental advantages, it has now become a viable alternative to concrete and steel for modern structures. However, fire is a major risk for wood structures. Therefore it is essential to have a substantial understanding of how wood behaves when subjected to a fire, especially when and how it will ignite. Ignition is a complex phenomenon which includes thermochemical processes in both solid and gas. A full numerical representation of ignition requires multiple complex calculations, making it impractical to solve ignition problems in most applications related to solid-phase response. One solution to this issue is to eliminate consideration of the gas phase, and instead adopt ignition criteria, which are values linked to thermal response parameters such as surface temperature and mass loss rate [1].

The majority of ignition studies in the literature use the assumption of a constant irradiation from a heat source. While this assumption is convenient due to its simplicity, it is singular and does not reflect a realistic fire scenario, for which irradiation tends to grow over time. Transient irradiation is a more comprehensive case, thus analyzing dynamic heat transfer effects and their impact on solid-phase ignition criteria is a relevant topic [2]. Previous studies have used transient heating scenarios for piloted ignition of different materials such as
polymers and wood. However, very few works look at spontaneous ignition under transient irradiation. DiDomizio et al. [3] evaluated a number of models in the literature that predict the spontaneous ignition of wood under time-varying exposure, but the current study is the first work that assesses the performance of solid-phase ignition criteria for wood under transient irradiation.

The main ignition criteria used in the literature for spontaneous ignition are the critical temperature, mass loss rate, heat flux, and time-energy squared. For a thermally thick solid, the critical temperature criterion (Eq. (1)) calculates the time to ignition from the assumption of the surface temperature at ignition and a constant irradiation [4]. This ignition criteria is the most commonly used in practice. The main limitation of this approach is that it cannot, by nature of its formulation, account for variations in external heat flux and environmental conditions [4]. The critical mass loss rate (MLR) criterion assumes that ignition takes place when a critical pyrolyzate mix with air flow of pyrolyzate mixes with air such that the mixture exceeds the lower flammability limit [5]. The most frequent problem with the critical MLR criterion is the difficulty in measuring mass loss prior to ignition [1]. The critical heat flux criterion represents the lowest value of constant irradiation under which flaming ignition is observed within an experimentally relevant time interval [6]. Finally, the time-energy squared correlation was developed by Reszka et al. [7], and it relies on identifying a relationship between the exposure time and the square incident heat flux.

\[
\frac{1}{\sqrt{q_{ig}}} = \frac{2}{\sqrt{\varepsilon k c_p}} \frac{q_{ig}}{T_{ig} - T_0}
\]

In this paper, all these criteria are evaluated for the spontaneous ignition of wood under transient irradiation by combining experimental measurements and a-priori predictions obtained from a one-dimensional model of pyrolysis. A-priori predictions are results of simulations run prior to performing the experiments, so they are not affected by bias [8]. They differ from a-posteriori predictions, which are the most used type of predictions in literature, that are done after the experimental measurements have been analyzed. In this paper, a-posteriori simulations are used as sensitivity analysis to show the influence of parameters such as heat of pyrolysis, reaction order, and radiative absorption coefficient.

### 2. Experiments

A series of experiments were conducted using a cone calorimeter apparatus on commercially available Canadian white spruce, a typical softwood used in construction. The specimens were conditioned to a 9% moisture content, and the wet density measured to be between 490 and 520 kg/m³. The sample size was 100×100 mm, with a thickness of 35 mm. For temperature measurements, 3 thermocouples were placed in the sample, located at 3 mm below the top surface, the middle of the sample, and 3 mm above the bottom surface. The thermocouples were stainless steel sheathed K-type (unexposed junction) with a 1.6 mm outer diameter. Three 1.98 mm diameter holes were drilled into one of the sides of the specimen at depths of 3, 19, and 35 mm. The instrumented specimen was placed on a 25 mm thick layer of ceramic fiber insulation, which was then placed on top of the standard cone calorimeter sample holder, filled with an additional 25 mm layer of insulation, as shown in the left side Fig. 1. This thick layer of insulation...
was used to ensure that the boundary condition on the bottom surface of the specimen could be approximated as adiabatic. To this end, an additional thermocouple was placed between the two layers of insulation in order to quantify heat losses through the bottom surface. The sample holder and sample were then wrapped in a layer of 25 mm thick ceramic fiber insulation on the outside, which was affixed to two locations with 20 gauge mechanics wire. Mass and temperature measurements were taken for 30 min in the case of no ignition or a few minutes after in case of ignition. As this study investigates spontaneous ignition of wood, the spark igniter was removed.

After the sample assembly was placed on the load cell, the cone heater was used to expose the top surface to a two-step transient irradiation. The cubic curve of the irradiation replicates a growing fire, which becomes steady after a certain time, at the threshold value. The transient irradiation scenarios were obtained by setting the initial temperature of the heater, initially ambient temperature, then increasing it using a digital temperature controller at a constant rate of approximately $1{\degree}C/s$ up to a setpoint value. The constant threshold value was applied until the experiments reached the 30 min mark, unless ignition occurred, in which case the experiment was stopped shortly after ignition. Five temperature setpoints were selected in this study, corresponding to steady-state irradiation values of 25, 27, 28, 30 and 50 kW/m². The threshold values of 27–28 kW/m² are of particular interest in the present work because ignition was less likely, but still possible, as seen in previous work [3] and indicated by the a-priori simulations.

Prior to the experiments, the transient irradiation scenarios were first characterized using a water cooled Schmidt-Boelter heat flux gauge. The cone heater temperature was also recorded, in both characterization tests as well as the experiments, to ensure that the transient heating profiles were identical in both cases. The irradiance over the surface of the specimen was uniform, although minor decreases towards the corners of the specimen were observed (less than a 5% decrease from the center value).

### 3. Pyrolysis model

Thermal decomposition of a wood specimen was modelled using the open-source code Gpyro [9]. The governing equations in a one-dimensional mass conservation (Eq. (2)), species conservation (Eq. (3)) and the energy equation (Eq. (4)). Thermal equilibrium between the condensed-phase and the gas phase is assumed. The shrinking of the sample was taken into account. As shown in the literature [10–12] in-depth radiation is important for wood for certain wavelengths, including those of the cone gauge. The cone heater temperature was also recorded, in both simulations.

The results of the a-priori simulations and experimental measurements are presented in Fig. 3 for the 30 kW/m² threshold and in Fig. 4 for the 50 kW/m² threshold scenarios. Because there were two repeats for each scenario, both experimental values are plotted, with an uncertainty cloud between them. In all of these four experiments, ignition occurred between 9 and 10 min or shortly after the 10 min mark. The temperature predictions show excellent agreement with the experimental measurements. Prior to the experiments, the transient irradiation scenarios were first characterized using a water cooled Schmidt-Boelter heat flux gauge. The cone heater temperature was also recorded, in both characterization tests as well as the experiments, to ensure that the transient heating profiles were identical in both cases. The irradiance over the surface of the specimen was uniform, although minor decreases towards the corners of the specimen were observed (less than a 5% decrease from the center value). The kinetic scheme used in the Gpyro model; each wood component (hemicellulose, cellulose, lignin) into active species (marked as (1) in Fig. 2), then each component decomposes into char and gases (marked as (2)) and tar (marked as (3)). This chemical scheme has been used frequently in the literature and has proven to be versatile in predicting a variety of experiments on wood materials. The kinetic parameters, namely the pre-exponential factor, activation energy and heat of pyrolysis for each reaction are given by Bellan [15] and were implemented in Gpyro by Richter [16]. They are summarized in Table 1.

Two sets of simulations were produced. A-priori simulations were done after the experiments were run, and before the experiments were run, thus avoiding bias. All 3 components of wood have the same thermal and physical properties. The thermal conductivity and specific heat capacity were taken at room temperature. Also, the model does not predict ignition, but simulates pre-ignition pyrolysis behavior. The ignition times are taken from the experiments and the model results after experimental ignition occurs are disregarded.

### 4. Results

The results of the a-priori simulations and experimental measurements are presented in Fig. 3 for the 30 kW/m² threshold and in Fig. 4 for the 50 kW/m² threshold scenarios. Because there were two repeats for each scenario, both experimental values are plotted, with an uncertainty cloud between them. In all of these four experiments, ignition occurred between 9 and 10 min or shortly after the 10 min mark. The temperature predictions show excellent agreement with the experimental measurements.
measurements, as presented in Fig. 3 and 4, being within the experimental uncertainty cloud. The MLR predictions are qualitatively similar to the measurement in the case of the higher irradiation case (50 kW/m²), however for the 30 kW/m², the measurement drops right before ignition, whereas the prediction shows an increase in mass loss rate, which is generally indicative of ignition. One of the explanations is that there was a lot of experimental uncertainty in the mass loss readings which were amplified in the differentiation to obtain the MLR.

For the scenario with a threshold at 25 kW/m², ignition did not occur in either of the repeats, thus the measurements were continued for a period of 30 min, as seen in Fig. 5. However, after around 10 min from the start of the experiment, the charring front passed the depth of 3 mm where the topmost thermocouple was placed. As this occurred the thermocouple dislodged from the charred surface, which means the recorded measurement was no longer representative. Therefore, the measurements at 3 mm depth were not relevant after 10 min.

Non-piloted ignition did not occur in the scenarios with a threshold of 25 and 27 kW/m² and in one repeat of the 28 kW/m² threshold scenarios and in the higher heat flux thresholds of 30 and 50 kW/m². The observed ignition times are summarized in Table 4. The ignition criteria analyzed are the critical surface temperature, MLR, heat flux, and time-energy squared.

### 5. A-posteriori predictions

The a-posteriori simulations were conducted after the experimental measurements were concluded and, while they do not contain any direct experimental results, they were done knowing what the experi-

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### Table 2

A-priori modelling parameters for wood, taken from [15] or measured.

| Property                        | Value | Units |
|---------------------------------|-------|-------|
| Thermal conductivity $k$        | 0.13  | W/m K |
| Density $\rho$                  | 490   | kg/m³ |
| Specific heat capacity $c_p$    | 2300  | J/kg K|
| Surface emissivity $\epsilon$   | 0.95  |       |
| Thermal conductivity of char $k_{char}$ | 0.08 | W/m K |
| Density of char $\rho_{char}$   | 330   | kg/m³ |
| Specific heat capacity of char $c_{p_{char}}$ | 1100 | J/kg K |

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![Fig. 3](image3.png) Fig. 3. Irradiation, temperature and MLR measurements vs. a-priori predictions for the scenario with 30 kW/m² threshold; experimental measurements are shown with uncertainty clouds, model predictions with solid lines; ignition occurred in this scenario.

![Fig. 4](image4.png) Fig. 4. Irradiation, temperature and MLR measurements vs. a-priori predictions for the scenario with 50 kW/m² threshold; experimental measurements are shown with uncertainty clouds, model predictions with solid lines; ignition occurred in this scenario.

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mental results look like.

The sensitivity analysis of the modelling was performed with an a-posteriori model. The parameters investigated are the heat of pyrolysis, in-depth radiation and reaction order. The scenario with a threshold of 50 kW/m² was used for presenting the sensitivity analysis, although the differences between this scenario and other are small (around 1%).

The heat of pyrolysis that was studied is the value used in the reactions from the kinetic scheme where the components of wood become char. In this chemical scheme [15] these reactions are exothermal, however, they are not suited for studying ignition. Therefore, the a-posteriori simulations reduced these values to zero and the results stay the same for all scenarios. That confirms the inadequacy of exothermic heat of pyrolysis for this fire application.

The in-depth radiation for wood depends on the radiation source. For wavelengths below 2 μm, corresponding to the wavelengths of tungsten lamps, in-depth absorption is negligible. For higher wavelengths, like the cone calorimeter at the temperatures in this study, the wood sample absorbs energy in-depth. The varying parameter for the sensitivity analysis is κ (m⁻¹), the absorption coefficient. As shown in Fig. 6 for the scenario using a threshold of 50 kW/m², a lower κ means higher in-depth absorption. The temperature value at 3 mm depth is very similar (4.3% increase) to the a-priori case without in-depth radiation, but there is significant variation (76% increase) in the temperature at 19 mm, as more energy is absorbed at a higher depth. For a higher κ, the temperature at 3 mm increases (21.8%) compared to the a-priori case, but at 19 mm the temperature does not increase significantly (5.0%). For both cases with in-depth absorption, MLR is higher, but for a lower κ, the increase in MLR starts later than for the other two cases, whereas for a higher κ the increase in MLR occurs at a similar time to the a-priori case. This shows that in-depth radiation should be taken into account when modelling cone calorimeter experiments.

The a-priori simulations had first-order reactions, as used in Bellan [15], therefore it is of interest to study the influence of the reaction order. Two different reaction orders from the literature are considered. This is marked as (a) in Table 3. The second value for the reaction order is taken from Huang et al. [18] and is marked as (b) in Table 3. Each reaction of the wood components decomposing into char has a different value: 2.4 for hemicellulose, 0.48 for cellulose, 10.4 for lignin. Fig. 7 shows that temperatures at the surface and at 3 mm depth decrease insignificantly (less than 1%) when the reaction order is not 1. Changes in MLR are more significant, increasing by 12.5% for case (a) and 7.5% for case (b), but the same shape of the curve is kept for all cases. Therefore the reaction order is an influential parameter for MLR, but it does not affect the temperatures in a significant way.

The same literature values for the reaction orders were used. The purpose of this study was to show whether or not the reaction order of the tar reaction is more influential than the char reaction. Fig. 8 shows that using a reaction order different from 1 for the tar reaction significantly changes temperature and MLR results. The surface
temperature is close to the temperature at 3 mm, which are lower and higher respectively compared to the base case where \( n = 1 \). The peak of the MLR curves is significantly higher when using a reaction order different from 1. This shows that the tar reaction is more influential than the char reaction.

### 6. Ignition criteria

Babrauskas [20] reviewed the first studies looking at the ignition criteria for spontaneous ignition of wood that did not involve oven-based experimental setups. These include Moran [21], Shoub and Bender [22], Melinek [23], and Abu-Zaid [24]. A few of the studies have calculated the surface temperature from correlations, thus being excluded from further analysis as well. There are a few more recent studies that have been included, namely Shi [25], Boonmee and Quintiere [26] and DiDomizio [3]. The most studied criterion is the critical ignition temperature, followed by the critical heat flux for ignition. Critical mass loss rate appears only in the newer studies and

| Property | Value | Source | \( e_{\text{prop},3\text{ mm}} \) [%] |
|----------|-------|--------|----------------|
| \( x \) | 129 | [19] | 4.3 |
| \( x \) | 570 | [12] | 21.8 |
| \( n_h(a) \) | 4.78 | [17] | 0.26 |
| \( n_l(a) \) | 4.78 | [17] | 0.26 |
| \( n_h(b) \) | 2.4 | [18] | 0.17 |
| \( n_l(b) \) | 0.48 | [18] | 0.17 |
| \( n_i(b) \) | 10.4 | [18] | 0.17 |

**Table 3**

Sensitivity analysis parameters; error was calculated for temperatures at a 3 mm depth as the difference between the a-priori results and the parametric study results.

![Fig. 7. Sensitivity analysis: influence of reaction order of char reaction (number 2 on Fig. 2) on temperatures at the surface and at 3 mm depth and MLR; \( n \) values taken from literature [17,18].](image)

**Fig. 7.** Sensitivity analysis: influence of reaction order of char reaction (number 2 on Fig. 2) on temperatures at the surface and at 3 mm depth and MLR; \( n \) values taken from literature [17,18].

| Irradiation Threshold [kW/m²] | \( \psi_{\text{crit}} \) [min] | \( T_{\text{crit}} \) [°C] | MLR<sub>crit</sub> [g/m²·s] | \( \dot{q}^{\prime}_{\text{em},i}$ [kW/m²] |
|-----------------------------|----------------|----------------|----------------|----------------|
| 25                          | 8.5            | 430            | 28             |
| 25                          | 6.5            | 430            | 28             |
| 27                          | 9.4            | 436            | 32             |
| 27                          | 4.5            | 436            | 32             |
| 28                          | 10.2           | 443            | 32             |

**Table 4**

Ignition criteria analysis for the non-piloted ignition of white spruce (surface temperatures are predictions, MLR and heat flux are measured).

**Fig. 8.** Sensitivity analysis: influence of reaction order of tar reaction (number 3 on Fig. 2) on temperatures at the surface and at 3 mm depth and MLR; \( n \) values taken from literature [17,18].

| Irradiation Threshold [kW/m²] | \( \psi_{\text{crit}} \) [min] | \( T_{\text{crit}} \) [°C] | MLR<sub>crit</sub> [g/m²·s] | \( \dot{q}^{\prime}_{\text{em},i}$ [kW/m²] |
|-----------------------------|----------------|----------------|----------------|----------------|
| 25                          | 8.5            | 430            | 28             |
| 25                          | 6.5            | 430            | 28             |
| 27                          | 9.4            | 436            | 32             |
| 27                          | 4.5            | 436            | 32             |
| 28                          | 10.2           | 443            | 32             |

**Table 5**

Maximum value of surface temperature, MLR and incident heat flux reached in the scenarios where ignition did not occur (surface temperature are predictions, MLR and heat flux are measured).
has a wide range of values.

Table 4 shows the scenarios where ignition occurred, as well as the measured values of MLR, heat flux and predictions of surface temperature at the time of ignition. Because the surface temperature was not measured, the model predictions of surface temperature are used in the analysis of ignition criteria. For all of the criteria, the values obtained in this work fit within the ranges found in literature. However, this is not indicative of the suitability of the criteria; since most of these values were obtained using a cone calorimeter or a fire propagation apparatus with constant irradiation exposures, it is not clear whether these values are applicable to other scenarios. Table 5 collects the maximum temperatures, MLR and irradiation reached in the scenarios where ignition did not occur. The maximum MLR in these cases is at least as high as the values recorded for the cases where non-piloted ignition occurred, and the heat flux and surface temperature are within the same ranges.

The most interesting case is the scenario with the threshold at 28 kW/m², where ignition occurred in four instances, but did not occur in one repeat. The heating conditions were the same (irradiation) and the sample behaved similarly (temperature & MLR); from this it must be concluded that neither of these criteria are able to predict the onset of ignition alone.

The final ignition criteria analyzed is the time-energy squared correlation, developed by Reszka et al. in [7]. This criterion requires a set of experiments for establishing the correlation between the exposure time and the energy squared. Then, the line of best fit of the correlation, which in the case of this set of experiments was a power fit, is compared with the exposure for every scenario and the intersection between the two lines is the predicted time to ignition. The predicted times to ignition are shown in Table 6. In the case of higher heat flux (50 kW/m²), the time to ignition is overpredicted by 17%, while for the 28 kW/m² it is underpredicted by 21%. The worst performance is for the 30 kW/m² scenario, where ignition is predicted to not occur. Thus, this criteria is not suitable for this set of experiments.

### 7. Conclusion

A one dimensional numerical model was developed and applied to study wood pyrolysis when subjected to transient irradiation. The model was validated using cone calorimeter experiments on Canadian white spruce which was exposed to a two-step transient irradiation scenario, the first part consisting of a growing irradiation followed by a constant threshold. These thresholds had a range between 25 and 50 kW/m². The experiments measured in-depth temperatures and mass loss rate, which were used to validate the model predictions.

The model used a chemical scheme with an initial reaction in which the virgin components of wood (cellulose, hemicellulose, lignin) become active, followed by the decomposition of each component into char and volatiles, and tar. The kinetic parameters, as well as the thermal properties of the wood were taken from literature for the a-priori model.

The surface temperature predictions of the a-priori simulations, the measured MLR and the measured heat flux were used in the analysis of the criteria for non-piloted ignition. While all the criteria are within the ranges of the values suggested in the literature, none can reliably predict if ignition will occur. Thus, the solid-phase criteria for non-piloted ignition are not easily applicable in cases with transient irradiation.

For the a-posteriori predictions, which were used as a sensitivity analysis, the heat of pyrolysis, reaction order and absorption coefficient were varied, with values taken from the literature. It was found that exothermic heats of pyrolysis do not have any influence on the predictions and the reaction order of the char reaction has an impact on the MLR (max. 12.5%), but it is insignificant on the temperatures (less than 1%). However, the reaction order of the tar reaction is more influential, increasing the peak MLR and changing the temperature results when it is different from 1. The in-depth absorption is an important factor in cone calorimeter experiments due to the wavelength of the radiation. For temperature predictions, the largest difference is a 76% increase in-depth and for MLR the differences are as high as 3 orders of magnitude.

For the first time in literature, the non-piloted ignition of wood under transient irradiation is studied with an emphasis on evaluating the performance of solid-phase ignition criteria. As these criteria have been established using constant irradiation scenarios, they prove to be inconclusive in predicting whether or not ignition will occur, showing similar values in all experiments. Therefore, there is a need to look into other indicators of ignition for the more general case of transient irradiation.

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