Thermal Diffusivity Measurements With Flash Method at Different Depths In a Burned Composite Material

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Abstract

The thermal diffusivity (TD) of burnt and unburnt composite materials is measured at room temperature and as a function of temperature (from 23°C to 338°C) [1, 2, 3, 4]. For each case at room temperature, the TD is calculated at the surface and after milling a 0.7 cm deep groove in each sample to observe the variations as a function of depth [5,6]. To obtain the TD at room temperature, the Parker's method is used. To obtain the TD as a function of temperature, the Parker's method is still used but with an oven to heat the sample and a laser diode to propagate a higher energy pulse in the sample [6].

1. Introduction

Infrared thermography can be applied in several areas and have many applications in non-destructive testing (NDT), medicine, arts, military and many others [3]. In NDT, one capability of infrared thermography is the measurement of thermal properties without destruction, for different types of materials. One such important thermal property for the characterization of a material is the thermal diffusivity (TD) [4]. Thermal diffusivity is a measure of the rate at which heat propagates through a certain unit area of a material. It can also be useful in the development of numerical models. There are several methods for measuring thermal diffusivity. The TD can be measured using different techniques such as steady state methods, quasi-steady states methods and transient methods [2,3]. As part of the transient techniques, Parker's method is commonly used for its speed and simplicity (in contrast to the other methods, transient techniques do not require to reach a permanent state to measure the TD) [2,3]. This paper includes results of TD of a burnt composite sample at room temperature and of an unburnt composite sample at room and elevated temperature (from 23°C to 338°C). The samples are made of carbon fiber reinforced epoxy and could be used to make lighter materials for applications in areas such as sport, aviation (reduced weight and fuel consumption), medicine and others. The results will be used to grasp how materials are affected after being exposed to a flame. On a wider scope, they will help develop numerical tools used to predict the reaction of composite samples subjected to compression and strain tests while being attacked by a flame. To obtain the TD at room temperature and elevated of temperature, the Parker's method [1] (also called the flash method) is used with a derivative technique call the partial time method [8]. At room temperature, with two heat lamps as excitation source and an infrared camera to observe the response of the material, the front face of the sample is briefly heated and the temperature evolution of the rear face is measured as a function of time. Infrared cameras are preferred to thermocouples as they are not in contact with the samples. From this temporal signal, the TD is calculated. To obtain the TD as a function of temperature, a different infrared camera is used while the sample is uniformly heated in an oven. For the transient excitation source, the two heat lamps are replaced by a laser diode to provide a heat wave of higher intensity in the sample. The paper shows first the mathematical principles used to calculate the TD. Secondly, we present in details the two experimental facilities used for the calculation of the TD at room and elevated temperatures. The results are then shown, analyzed and compared with TD data available in the literature, to reveal the coherence with others studies on carbon fiber material. Finally, relevant conclusions are presented.

2. Principles

After an transient thermal excitation of the sample with a pulse of energy (flash lamps, laser ...), the rising temperature at the rear face that can be observed by using different techniques (infrared detector, infrared camera...). Figure 1 shows the general scheme. The solution to the one-dimensional equation of thermal conduction, with the assumption of ideal conditions, describes the temperature on the rear face of a sample as [1]:

$$\Delta T = \Delta T_m \left[1 + 2 \sum_{n=1}^{\infty}(-1)^n \exp\left(\frac{-n^2\pi^2\alpha t}{L^2}\right)\right] \tag{1}$$

where ΔT represents the elevation of the temperature of the sample, ΔT_m is the maximum value for the temperature, L is the thickness of the specimen and α is the thermal diffusivity.
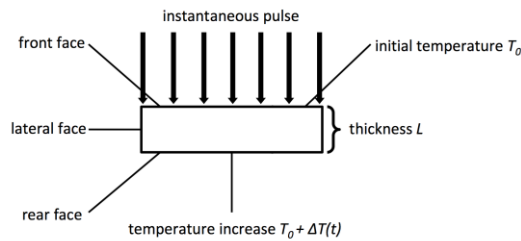

Fig. 1 General scheme for the heat transmission from the front to the rear face of the sample with the flash method

Two dimensionless parameters are introduced, V and ω to represent respectively a dimensionless measure of temperature and time. They are defined as:

$$V = \Delta T / \Delta T_M \tag{2}$$

$$\omega = \frac{\pi^2\alpha t}{L^2} \tag{3}$$

The combination of equations 1, 2 and 3 yields an expression for the evolution of the dimensionless temperature at the back face as a function of the dimensionless time:

$$V = \left[1 + 2 \sum_{n=1}^{\infty}(-1)^n \exp(-n^2\omega)\right] \tag{4}$$

Figure 2 shows an example of this evolution obtained from equation (4), showing that the backside temperature asymptotically reaches a maximum steady-state value following the application of the transient heat excitation. This figure, called a thermograph, shows the evolution of the temperature of the material's rear face as a function of time. It is important to specify that figure 2 shows a theoretical thermograph where heat losses are neglected. Even though, steady state is attained on this figure, Parker's method does not require a steady state in order to rightfully measure a material's TD. Obviously, in materials with higher thermal diffusivity will reach this steady state temperature faster.
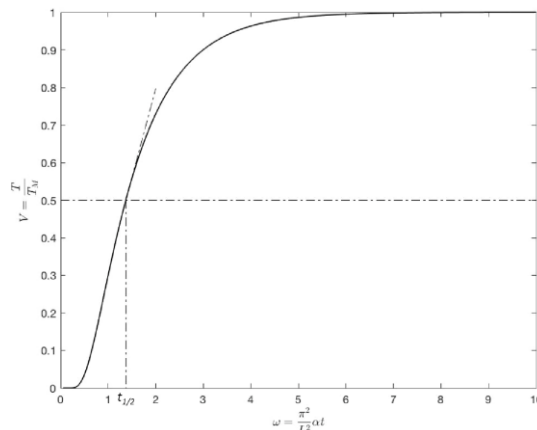

Fig. 2 Theoretical thermograph for the flash method

The thermal diffusivity, α, can then be calculated using two standard approaches. The first considers the point when V equals 0.5 and when w equals 1.38:

$$\alpha = \frac{1,38 \, L^2}{\pi^2 t_{1/2}} \tag{5}$$

where $t_{1/2}$ is defined as half the time to reach the maximum temperature.

One can also draw an extrapolated straight line from the initial temperature rise at the back face that intercepts the time axis at t_x, as sown in figure 2. At this point w=0,48 which yields another relation:

$$\alpha = \frac{0.48 \, L^2}{\pi^2 t_x} \tag{6}$$

In practice, when the TD is calculated with the flash method, there are thermal losses that are inevitable following the excitation of the sample. Many mathematical models have been proposed to calculate the TD by doing restrictive assumptions about the extent of the heat losses [8-11]. One method has however been developed that does not require restrictive assumptions about the heat losses. This method is called the partial time method or Degiovanni method [10]. This method is based on the temporal momentum equation of order 0 and -1. The partial time method has the advantage of keeping the simplicity of the Parker's method and also to keep the important part of the thermograph as shown in figure 3 (The important part of the thermograph is the section of the time where we have the maximum and the minimum temperature of the rear face after excitation of the front face). When using this method, three intermediate TDs are calculated: $\alpha_{1/3} , \alpha_{1/3} , \alpha_{2/3} for$ the materials, based on the times corresponding to 1/3, ½ and 2/3 of the maximum temperature, as follows:

$$\alpha_{\frac{1}{3}} = \frac{L^2}{t_{\frac{5}{6}}} \left[0,818 - 1,708 \frac{t_{\frac{1}{3}}}{t_{\frac{5}{6}}} + 0,885 \left(\frac{t_{\frac{1}{3}}}{t_{\frac{5}{6}}} \right)^2 \right] \tag{7}$$

$$\alpha_{\frac{1}{2}} = \frac{L^2}{t_{\frac{5}{6}}} \left[0,954 - 1,581 \frac{t_{\frac{1}{2}}}{t_{\frac{5}{6}}} + 0,558 \left(\frac{t_{\frac{1}{3}}}{t_{\frac{5}{6}}} \right)^2 \right] \tag{8}$$

$$\alpha_{\frac{2}{3}} = \frac{L^2}{t_{\frac{5}{6}}} \left[1,131 - 1,222 \frac{t_{\frac{2}{3}}}{t_{\frac{5}{6}}} \right] \tag{9}$$
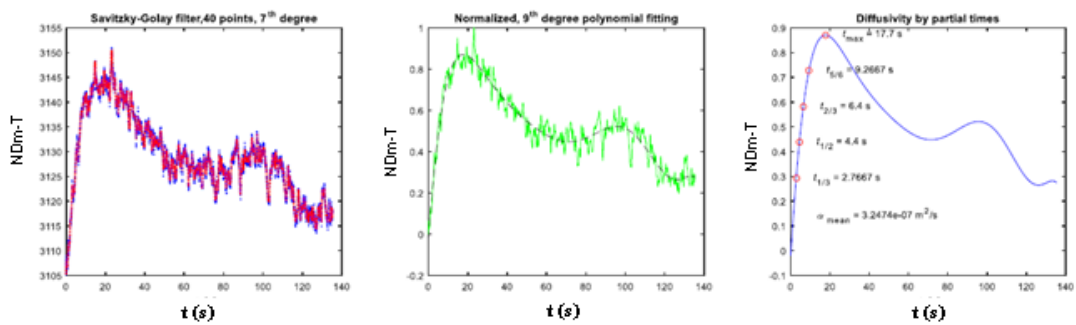

Fig. 3 Thermographs for the partial time method
(Even though the thermograph does not reach a steady state, it still shows a temperature rise and the attainment of a maximum in the transient state. As previously stated, Parker's method and Degiovanni's method do not require to reach a steady state in order to measure the TD.)

3. Experimental facility

Two different setups were developed to calculate the thermal diffusivity: one for the TD at room temperature (figure 4a) and another that allows the evaluation of the TD as a function of temperature (figure 4b). In both cases a pulse of energy is sent to a composite sample made of carbon fiber reinforced polymer. A thermal wave front is then transmitted through the surface of the sample and allowed to propagate below the surface, as described by the equation of diffusion [2]. The pulse of energy can be generated by flash lamps (figure 4a), or a laser beam (figure 4b) as well as by water or air jets [3]. The pulse of energy can be either hot or cold because it just needs to generate a difference in temperature through the sample. The sample is then analyzed during the rise or fall in temperature. The flash method has the advantage of being easy to use in addition and of quickly evaluating the TD, typically in less than a minute for carbon fiber reinforced composites.
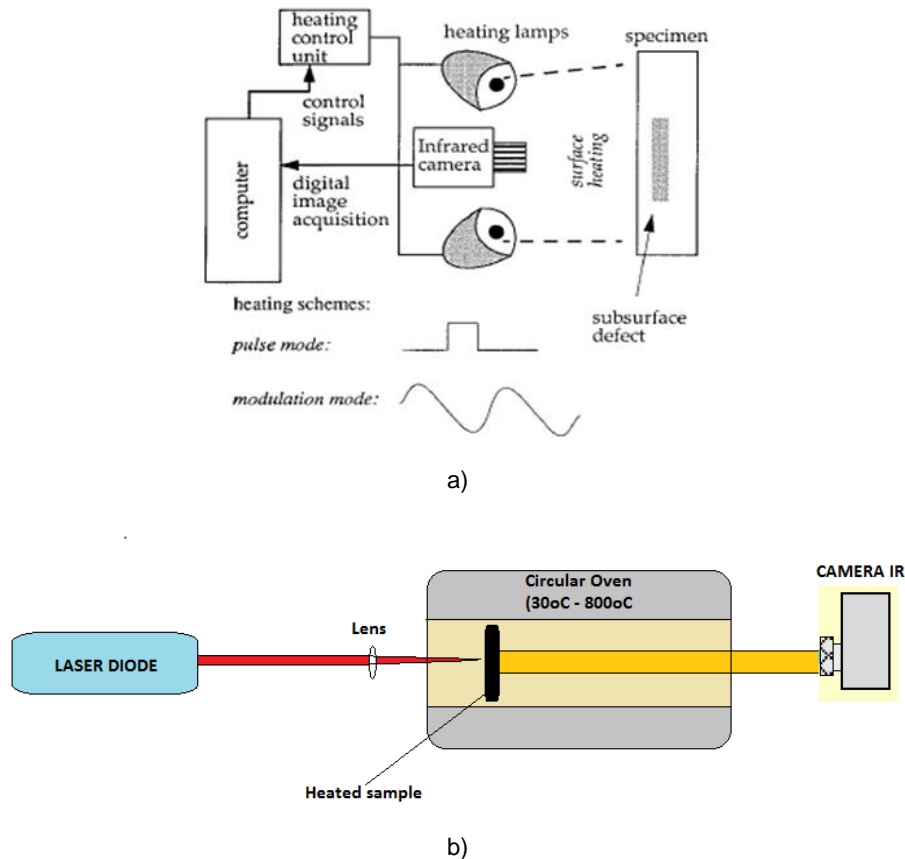

a)

b)

Fig. 4: a) *Experimental setup used for the measurement of thermal diffusivity at room temperature [3] and b) as a function of temperature*

3.1 Thermal diffusivity at room temperature

Figure 4a shows the setup used to measure the thermal diffusivity. To stimulate uniformly the samples, they are briefly heated with two flash lamps (6.2 kJ/flash, 2 ms heating duration). An infrared camera (InSb, 3-5 µm, 640x512 pixels) is used to view the rise in the backside temperature of the samples and an in-house software calculates the TD using an algorithm in MatLab based on eq. 1 & 2. For the Parker method, a uniform pulse of energy in space and time is needed, resulting in a small rise in temperature at the rear face. The duration of the pulse should be adjusted, from microseconds to seconds to achieve the desired temperature rise, depending on the thickness of the material and its thermal properties. The carbon fiber samples under investigation here have a thickness between 1cm and 2cm. An example of a temperature variation gathered in such a sample is shown in figure 5, with the theoretical model providing similar results. The temperature rise following pulse excitation can be divided in two phases: first an initial increase to the maximum temperature, followed by a slow cooling phase. From the thermograph presented in figure 5, only the initial rise in temperature and the maximum temperature are important to calculate the TD.
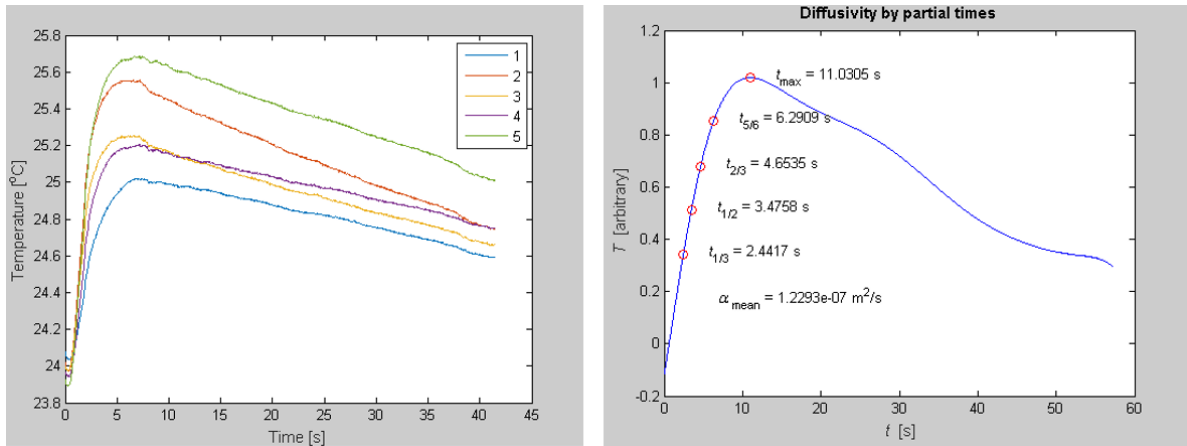
Fig. 5 Examples of rising temperatures recorded on the back side of the sample, used in the partial times method

3.2 Thermal diffusivity as a function of temperature

This section describes the set up to measure the TD as a function of temperature. For this purpose, a high temperature tubular oven is used to heat the samples, as illustrated in Figure 4b. In this case, instead of using flash lamps to simulate the samples, the samples are briefly heated by a laser diode (808nm, 980ms, 30W). A different camera (InSb, 3.97-4.01 µm, 640x512 pixels) is used to observe the radiation at high temperature. The same algorithm is used to measure the TD from the experimental data [6]. Figure 6 presents an image of the sample and the hot spot created by the laser diode as seen through the oven. The curves obtained using this method are similar to those at room temperature (a steep initial rising phase followed by the maximum temperature and a slow decay in temperature).
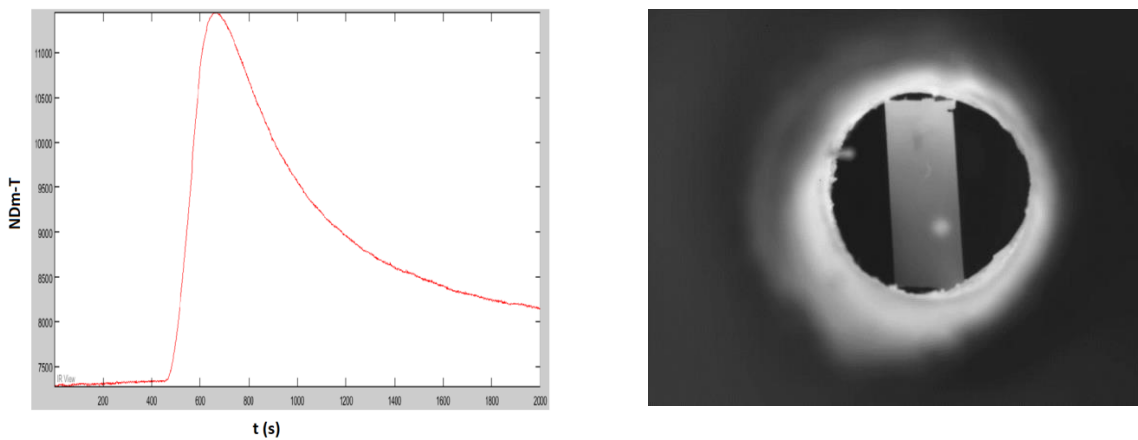

Fig. 6 a) Infrared image of the composite sample in the cylindrical oven, with the hot spot created by the laser pulse. b) Evolution of temperature as a function of time

4. Results

The results for the TD measured for different composite samples are gathered in Table 1. Those samples are rectangular as shown in figure 8 and are of 25cm in length and 2.5cm in height and vary in thickness from 1mm to 2mm. First, the TD for different areas of two burnt composite samples is presented in column *a* of the table (without and with a milled groove) [4]. Figure 8 shows one of the two burnt composite samples without and with a milled groove. The milling of this groove on burnt samples allows some of the partially charred material to be removed, yielding a measurement of the thermal properties of the less degraded material below. Second, the TD of the six unburnt samples is shown in column *b* of the table. Third and last, in column *c*, the TD for one sample at different temperatures is shown. These results are in qualitative agreement with previous works as reported in [7], in which the samples are Carbon Fiber Reinforced Polymers (CFRP) with a thickness varying from 1.02 mm to 5.2mm. The results show a TD of 3.72e-07 m²/s using a x-ray beam.

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Table 1. *a) Thermal diffusivity measured on two different partially burned composite samples. Area label #1 is on the burned region and areas #2 to #5 are over unburned regions (figure 7). b) Results for different unburnt samples. c) Results for the first unburnt sample as a function of temperature.*

a					b		c	
Area	Thermal Diffusivity of 1st burnt sample without Milled Groove [m²/s]	Thermal Diffusivity of 1st burnt sample with Milled Groove [m²/s]	Thermal Diffusivity of 2nd burnt sample without Milled Groove [m²/s]	Thermal Diffusivity of 2nd burnt sample with Milled Groove [m²/s]	Samples	Thermal Diffusivity of Unburnt samples [m²/s]	Temperature [°C]	Thermal Diffusivity of Unburnt sample as a function of temperature [m²/s]
1	1.23e-07	2.40e-07	2.10e-07	2.22e-07	1	2.69e-07	23	2.01e-07
2	1.83e-07		3.07e-07		2	3.83e-07	46	2.21e-07
3	2.03e-07	2.32e-07	3.14e-07	2.41e-07	3	2.57e-07	75	2.89e-07
4	1.92e-07	2.65e-07	2.74e-07	2.25e-07	4	3.43e-07	100	2.49e-07
5	2.00e-07		2.89e-07		5	2.89e-07	150	9.48e-08
					6	3.61e-07	189	1.28e-07
							230	1.53e-07
							338	1.05e-07

4.1 Thermal diffusivity at room temperature

4.1.1 Results for unburnt composite samples

The TD was first measured over area corresponding to each pixel of the images acquired by the infrared camera, both on intact samples and after the milling of a 0.7 mm deep groove. A map of the TD values calculated is shown in Figure 7, with a spatial resolution sufficiently fine to observe the effect of the reinforcement carbon fiber fabric (which has a much higher thermal diffusivity than the polymer matrix). For the six unburnt samples evaluated, we observed a variation of the local TD between 2e-07 m²/s and 6e-7 m²/s, while the average values varied much less, as shown in column *b* of Table 1. The thermal diffusivity maps shown in figure 7 reveal that when the grooved is milled and the sample becomes thinner, the inhomogeneity in the distribution of the TD from the effect of the carbon fiber fabric increases.
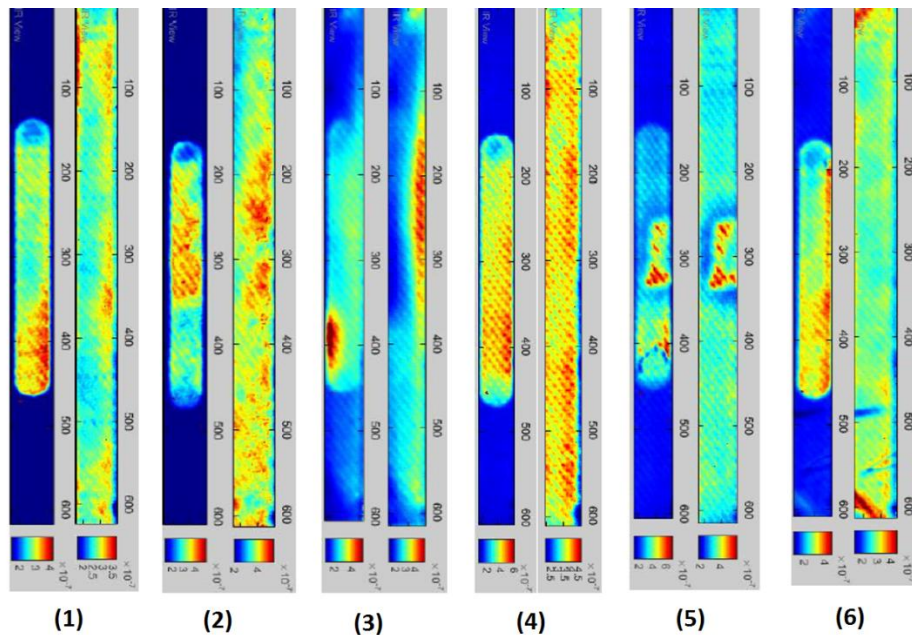

(1)　(2)　(3)　(4)　(5)　(6)

Fig. 7: *Maps of the thermal diffusivity (TD) for six unburnt composite samples. For each samples, the 1st map represent the TD before milling a groove, and the 2nd map of each set is the TD after milling a 0.7 mm deep groove.*

4.1.1 Results on burnt composite samples

The mean TD is measured for two different partially burned composite samples, either over their full thickness or after a 0.7 mm deep groove has been milled in the material. These samples were exposed to a propane flame for 15 minutes. We measured the mean TD over the sample thickness by selecting different areas on the samples. Figure 8 shows the first partially burnt sample, both with and without the milled groove. For the first burnt sample, the mean TD over an area affected by the flame is 1.229e-07 m^2/s. After the top layer of charred material has been removed, this value increases to 2.405 m^2/s. For the second partially burned sample, these values are 2.103e-07 m^2/s and 2.218e-7m^2/s, respectively. These high differences between samples reveal the high variability in the process used to expose the samples to the flame. However, in the first burnt sample, the measured TD increases with depth over burnt areas. This indicates that the charred layer at the surface of the samples exposed to the flame has a lower thermal diffusivity than the virgin material. For the second sample, results show the opposite. This may be due to the flame reaching the backside of the sample and in turn modified its composition. It must be noted here that these results can be affected by milling machine used. Certainly, dust is created by using the milling machine that fills the pores of the material. Depending on the properties of the dust formed, this can affect the measurement.
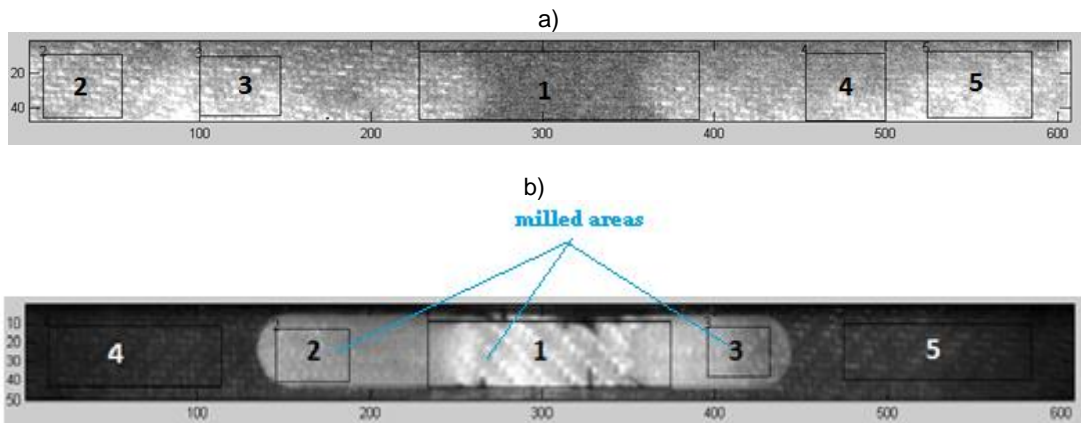

Fig. 8: *Pictures of the selected areas of the 1st burnt sample (a) without milled groove, (b) with milled groove (0.7mm deep)*

4.1.3 Discussion

The thermal diffusivity at room temperature of two types of composite materials is obtained based on the parker's and partial time method. Those techniques have an advantage to be non-destructive when the thermal diffusivity is calculated at the surface. The results presented here show the possibility to produce a map of the TD with high spatial accuracy over intact and partially burned samples.

The first type of composite material the six unburnt samples, a variation of the TD is observed between 2e-07 m^2/s and 6e-7 m^2/s. This variation mainly depends of the material composition of each sample and the dust produced after the use of the milling machine that contaminated the sample. In the next experiences with the samples air-blowing will be use to see the difference before the calculus of TD.

The second type of composite material was burnt and the second was unburnt. The TD is calculated at the surface and then deeper in the material. The thermal diffusivity for those with a burnt area increases with depth. For the unburnt samples, in this part, the TD is measured for each pixel first at the surface and at a 0.7 mm deep groove milled in the material. The mapped TD values are calculated at each pixel as shown on Figure 8.

4.2 Thermal diffusivity as a function of temperature

4.2.1 Results and discussion

The thermal diffusivity (TD) of unburnt composite samples is measured as a function of temperature using Parker's method associated with the partial time method. An oven is used to heat the sample and a pulse from a laser diode permits to briefly increase the temperature of the heated sample. A high temperature infrared camera records the quick rise of the radiation following the laser pulse to extract the TD values. Column *c* in Table 1 summarizes the results of the TD as a function of temperature for the unburnt sample, these results are reproduced in figure 9. From 23°C to 75°C, TD increases monotonically from 2.01e-7 m^2/s to 2.89e-7 m^2/s and then decreases to reach 2.496e-7 m^2/s at 100°C. Between 150°C and 330°C, the TD increases again then decreases with another maximum of 1.53e-7 m^2/s at 230°C.
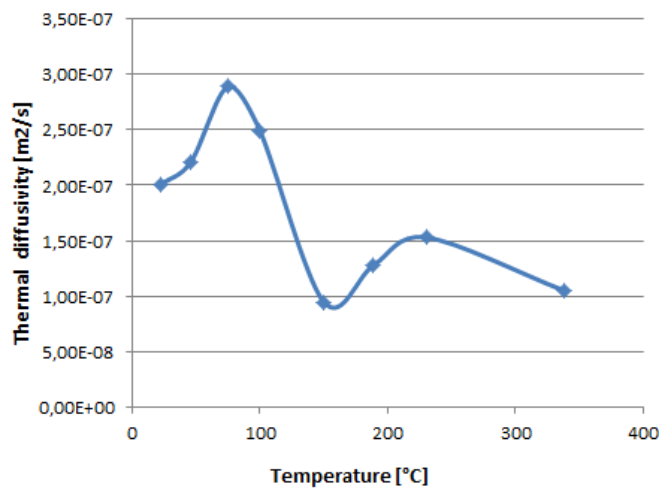

Fig. 9: *Thermal diffusivity as a function of temperature for an unburnt sample carbon fiber reinforced composite*

5. Conclusion

This paper presents the measurement of the thermal diffusivity of different samples of carbon fiber reinforced composites. The TD is measured both over the whole thickness of the material and over a reduced depth after a grooved has been milled in the sample. Measurements are carried out with partially burnt and unburnt samples at room temperature and at elevated temperature. The results show that Parker's method is adequate for such measurement when there is not much losses of thermal energy. Degiovanni's method (or partial time method) is convenient if the material could be expected to dissipate a significant amount of heat rather than simply transmit it one-dimensionally across its depth. The different TDs obtained are in agreement with values available obtained from the literature for similar materials [7]. Another way to obtain the TD could be the use of cold air jet flow to compare with the different results at room and as a function of temperature. Moreover, to avoid the use of the milling machine to measure in-depth, one could calculate the TD using the quadrupole method [12] This will be tried possibly in the future, if time will permit.

Table 2. Summary for each of the TD measurements in this article

Room temperature				High temperature	
Unburnt		Burnt		Unburnt	Burnt
Surface	Depth	Surface	Depth	Surface	

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