

DEVELOPMENT OF AN EXPERIMENTAL BENCH FOR ANALYSING HEAT TRANSFER DURING THE TAPE PLACEMENT OF CARBON/PEEK COMPOSITES

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ABSTRACT

An instrumented bench is developed for the accurate measurement of the heat transfer occurring during the manufacturing of composite parts by Automated Fiber Placement. It aims at being representative of the different steps of the process which are: the heating of the deposited pre-impregnated tape and of the substrate, the creation of a new interface by bringing them quickly into contact and its consolidation by cooling. After the complete characterization of the laser output (the spatial distribution of the outgoing flux and the calibration of the laser power), different process configurations are tested by varying the laser inclination and power. The thermal contact resistances between a tape and the stainless steel bench, and between two tapes, are of particular interest. They will be assessed thanks to a specific metrology composed of thermocouples and heat flux sensors, as well as suitable inverse methods. The data collected will be compared to a finite element 2D model of the Automated Fiber Placement process.

1 INTRODUCTION

The Automated Fiber Placement process (AFP) is nowadays a generalized technology for processing carbon fibers polymer composites in the aerospace industry [1]. The improvement of advanced thermoplastic polymers, which are now more mechanically efficient and resistant to extreme environments than the commonly employed thermoset counterparts, offers the possibility of an enhanced and faster process [2]. As a matter of fact, major productivity gains could be reached thanks to a reduced post-consolidation time, currently achieved with a costly autoclave step [3]. Yet, a fine understanding of the evolution of the different kinetics of the thermally-activated involved phenomena, is required, according the process parameters, during the composite consolidation [4]–[6].

Indeed, in this process, a laminate is manufactured by the stacking one upon another with the help of motorized placement head, of tapes pre-impregnated with unidirectional carbon fibers and matrix. At each deposition pass, the incoming tape and the substrate are heated up together, with a near infrared laser diode, and put into contact by a compaction roller that applies a given pressure (Figure 1). In the case of thermoplastic matrix, thanks to the ability of the crystalline parts to melt, the bonding at the interface is achieved by the inter-diffusion of the polymer chains from each side [7]. This mechanism known as autohesion [8] triggers the development of an interfacial strength, guarantee of the bond quality, and is dependent of the molecular weight of the polymer but also of time, temperature and pressure. However, the bond healing can occur only where two melted polymer rich zones are effectively in contact, what depends on their respective topology. With the help of the applied temperature and pressure, the polymer viscosity decreases thus improving the ability of the surfaces to accommodate. A so-called intimate contact then develops at the contact zone under the roller [9]. When the pressure is released, the newly formed laminate surface can cool, generally under

room conditions, before the next pass. Other temperature and pressure dependent phenomena such as the crystallization of the polymer matrix [10], the release of residual stresses [11], some void growth inside the tapes and at the interface [12], and most of all the polymer degradation [13] will influence the final quality of the bond. Indeed, if the main criterion for triggering autohesion at the interface is to be above the melting point of the polymer, the surface temperature of the substrate and tape prior to contact must stay below the degradation threshold of the polymer as well. In the case of a PEEK (PolyEtherEtherKetone) matrix for instance, the target temperature window is then defined between 343°C and 500°C [13]. The upper limit also decreases for lower speed of lay-up. Moreover, due to the inclination and the curvature of the deposited tape prior to the nip-point (see Figure 1), a shadow zone appears where the tape and substrate are not illuminated by the laser before contact. The temperature drop in that area is not easily modelled nor controlled. From there, it appears that the consolidation is very sensitive to the thermal field evolution. In order to find an adequate process window the thermal and pressure history must be accurately known according to the process conditions.

Since direct temperature measurements on the process and the exploitation of such recorded data are uneasy [14]–[16], the temperature field is often predicted. A comparison is then made with the mechanical strength of testing samples manufactured in the same process conditions. Heat transfer occurring during the heating step is then still to be measured. Stokes-Griffin [14] and Groupe [16] attempted to measure the surface temperature of the tape and substrate with the help of infrared camera. In both experiment the temperature profiles were over predicted due to the reflection of the flux emitted by the surfaces facing each other. Also, examples of contact temperature measurements between the prepreg plies during the placement of new tapes with thermocouples can be encountered [15]–[20]. In addition to the mentioned arduous instrumentation, questions concerning the intrusiveness of the sensors between the thin composite tapes and the exact meaning of the measured temperatures are often let aside.

This study offers to step aside from such difficulties by developing a specific bench aiming to experimentally determine what prepreg tape and substrate undergo during deposition in terms of temperatures and heat flux fields. The modular device consists in two instrumented parts that will allow measuring the thermal quantity of interest during the AFP process such as the thermal contact resistances and the surface temperature.

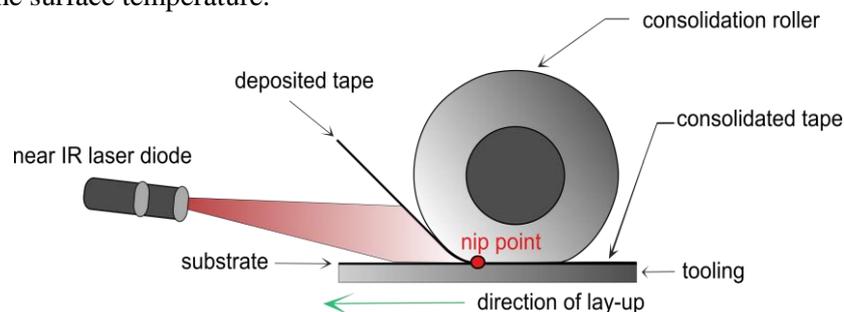


FIGURE 1: Schematic representation of the Automated Fiber Placement process

2 MATERIAL AND METHODS

2.1 DESCRIPTION OF THE INSTRUMENTED BENCH

2.1.1 Configuration n°1

The first step of this ongoing work consists in a fine study of the interaction between a laser diode and the composite according to the laser inclination and power (Figure 2).

The metallic part consists in a static stainless steel bench of 20*100*12 mm³ dimensions. The bench has been design such as heat transfer is 1-D through its thickness. It is instrumented with 21 K-type 50µm diameter thermocouples welded at 250 µm from the surface, with one central heat flux sensor [21], composed of three coaxial thermocouples, and with lateral control thermocouples. As can be seen

on Figure 2 zoomed inset, the locations of the thermocouples in the right and left sides of the bench are shifted from 2.5 mm. Moreover, the lower part of the bench can be heated up to 400°C and the surface temperature is measured with the help of a FLIR SC7000 (InSb) infrared camera aligned with the flux sensor axis. Finally, the lateral sides are thermally isolated in order to master the thermal fields within the part.

Carbon fibers/PEEK composite tapes of dimensions 12* 60 mm² are maintained on the surface of the bench with the help of 50 N magnets and are heated by a laser diode Laserline 2 kW. The inclination of this latter can be varied from $\theta = 0^\circ$ to 70° with respect to the normal axis to the surface when the infrared camera is removed. The minimum angle reaches 3° otherwise.

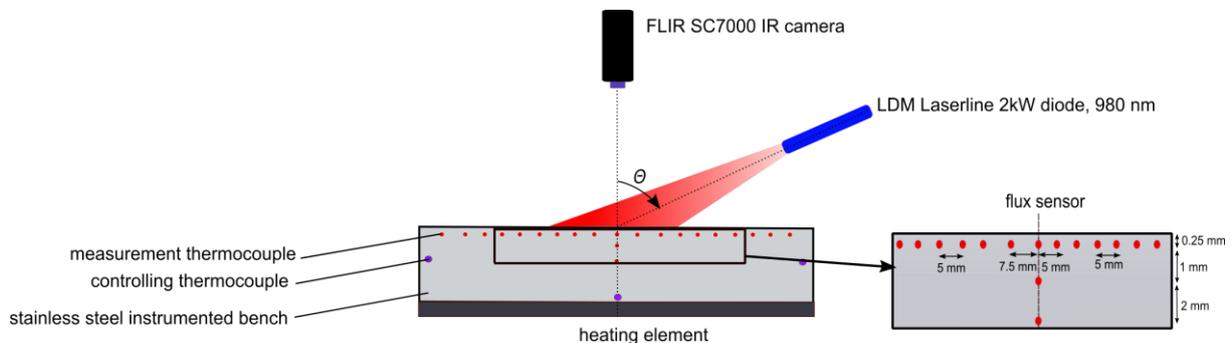


FIGURE 2: Presentation of the instrumented bottom part

Thanks to the fine metrology and the use of appropriate inverse methods, the thermal contact resistances between the mold and a tape and between several layers of a carbon fiber laminate will be measured.

2.1.2 Configuration n°2

A top dynamic part similarly instrumented can be set-up on the bench from Configuration n°1. The complete system aims at being representative of the different steps of the process which are: the heating of the deposited pre-impregnated tape and of the substrate, the creation of a new interface by bringing them into contact and its consolidation by cooling. The tapes will be then be heated, put in contact and let cooled by air after a quick back and forth motion of the upper part (see Figure 3). The motion of the upper part is controlled by a solenoid valve.

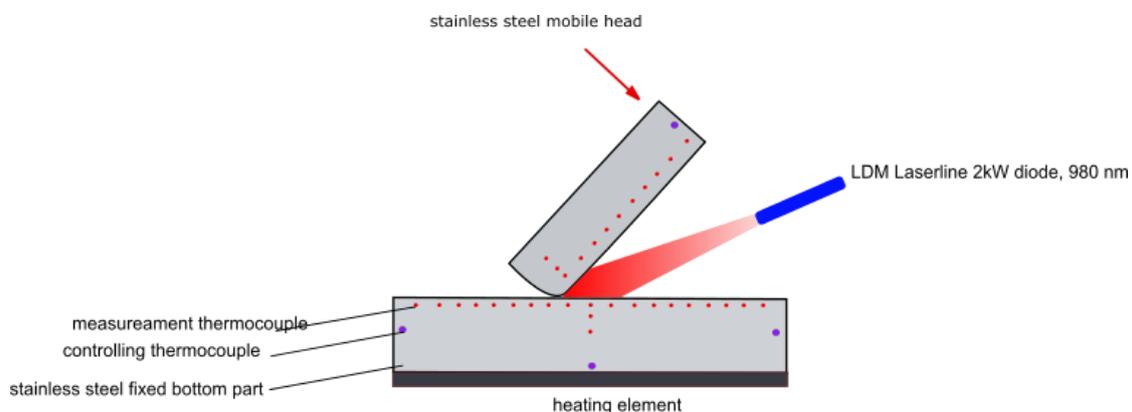


FIGURE 3: Presentation of the complete set-up.

2.1.3 Heat flux sensor

As mentioned in 2.1.1 and 2.1.2 sub-sections, flux sensors are located in the bench. One in the fixed and one in the moveable part of the bench, respectively. They consist in three type K-thermocouples TC1, TC2 and TC3, welded in the stainless steel bulk on the same vertical axis. As described in Figure 2, their respective locations with respect to the bench surface are 250 μm, 1.25 mm and 3.25 mm.

When the flux sensor is crossed by one dimensional flux, the magnitude of the incoming flux at the surface of the bench can be calculated with the help of the Beck inverse method [22].

2.1.4 Characterization and calibration of the laser diode output

The 2 kW Laserline near infrared laser diode used in this study is equipped with an optical system that transforms the output of the optical fiber plugged to the diode into a $12 * 12 \text{ mm}^2$ square at a focal distance of 150 mm. The flux density along the laser spot has a top hat shape where its magnitude sharply decreases at the spot edges. The output being divergent, the flux distribution must be characterized according to the distance between the laser and the bench surface and to the angle of incidence.

The laser diode output is controlled in terms of a percentage of its maximum magnitude available. The power density in the center of the laser spot was calibrated using the central heat flux sensor from the fixed bottom part. To do so, the surface of the bench was painted with a high emissivity coating (referenced as Black Velvet 821-11). The infrared camera was removed and the laser arm was adjusted in the position $\theta = 0^\circ$. The maximum flux magnitude for a 100 % selected power was measured to be 18 MW/m^2 .

Then, the divergence of the laser beam was determined using the visible pointer of the laser diode. Since the visible beam goes through the same optical path inside the shaping system, its final distribution is similar. The distance between the opposite edges of the visible square were measured on a target placed at different distances from the optical system (Figure 4). The final angular divergence of the beam was evaluated to 2.12° .

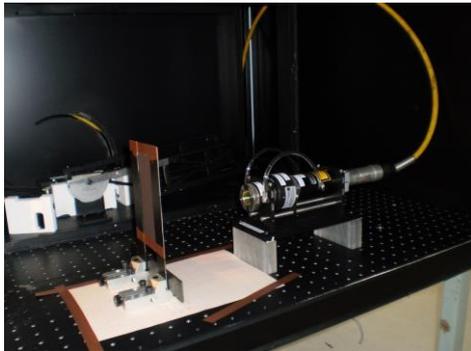
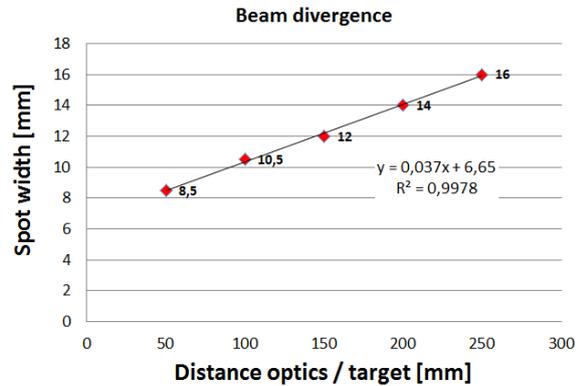


FIGURE 4: Experimental set-up for the laser divergence measurement



Finally, the spatial distribution of the laser diode was characterized using a radiometric method similar to the one used in [23]. A 3 mm thick stainless steel sheet was covered with the high emissivity coating and installed on the bench (Figure 5). The laser beam was inclined with an angle $\theta = 3^\circ$, which is the minimum angle available when both the laser and infrared camera are used. The laser was tuned on 10 % of its power and the surface was irradiated during 50 ms and the temperature evolution of the surface was recorded with the help of the infrared camera. The operation was repeated every 10° up to an incidence $\theta = 70^\circ$.

During the first instant of the heating, the lateral diffusion can be neglected and the temperature profile is proportional to the power density profile.

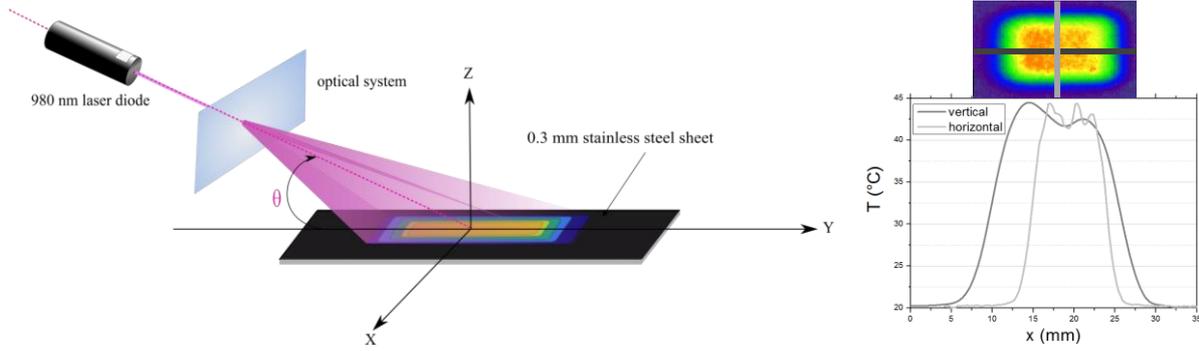


FIGURE 5: Experimental set-up for the characterization of the laser flux distribution; On the left; Schematic of the set-up, On the right, example of temperature profiles measured in the lateral directions.

By normalizing the lateral temperature profiles, the following law is obtained to describe the normalized flux distribution according to the x and y axes:

$$\hat{q} = \frac{1}{1 - \frac{Y}{L} \cos(\theta)} \exp \left(- \left(\left(\frac{X}{\sigma(Z)} \right)^n + \left(\frac{Y \sin(\theta)}{\sigma(Z)} \right)^n \right) \right)$$

In this equation, θ is the inclination of the laser with respect to the normal axis, Z is the laser optics / target distance, L is the spot size for $\theta = 0^\circ$, and X and Y are the coordinates of a point of the target as indicated on the graphic above. σ is the standard deviation of the distribution that characterizes the width of the power distribution inside the spot for $\theta = 0^\circ$, and n is an integer that defines the sharpness of the distribution edges. After analysing the profiles, we found that $n = 8$ and $\sigma = 0.005$.

2.2 MATERIAL APC2

The material used in this study is a composite called APC2 provided by Cytec. It consists of a matrix PEEK (PolyEtherEtherKetone) reinforced by unidirectional carbon fibers. Its average thickness was evaluated at $150 \mu\text{m}$.

The thermal properties used for modelling in the composite thickness are summed up in the following table, where T is the temperature:

TABLE 1: Material Properties for the Fibers, Peek Matrix and Composite

Properties	Value
Fiber mass fraction %m	0.76 (supplier data)
Fiber volume fraction %v	0.55 (supplier data)
APC2 density [kg/m³], ρ	1550
Transverse thermal conductivity, k [W.m⁻¹.K⁻¹]	0.43 (experimental measurement)
Heat capacity PEEK [J.kg⁻¹.K⁻¹], $C_{p,PEEK}$	953.4+3.103*T[K] (DSC measurement)
Heat capacity fibers, $C_{p,Fibers}$	577.4+6.851165*T [K]-0.018078*T² [K²]
Heat capacity APC2, $C_{p,APC2}$	(1-%m)*$C_{p,PEEK}$+%m*$C_{p,Fibers}$

2.3 THERMAL CONTACT RESISTANCE CALCULATION METHODE

Let us consider an APC2 tape maintained in contact on the stainless steel bench surface. The thermal contact resistance between the tape and the mold is defined as the difference of temperature from each side of the interface on the flux crossing the interface:

$$R_{tc} = \frac{T_{interface,+} - T_{interface,-}}{\varphi_{out}}$$

If the surface of the composite is irradiated by a flux φ_{in} the surface temperature is recorded with the help of the camera and the flux φ_{out} is measured with the help of the heat flux sensor. The temperature $T_{interface,-}$ is then easily calculated.

A 1D continuous model of the tape is then implemented with the boundary condition: $T_{surface}$ for $x=0$ and φ_{out} for $x=150 \mu\text{m}$. The evolution of $T_{interface,+}$ is thus obtained numerically and allow the calculation of the thermal contact resistance RTC.

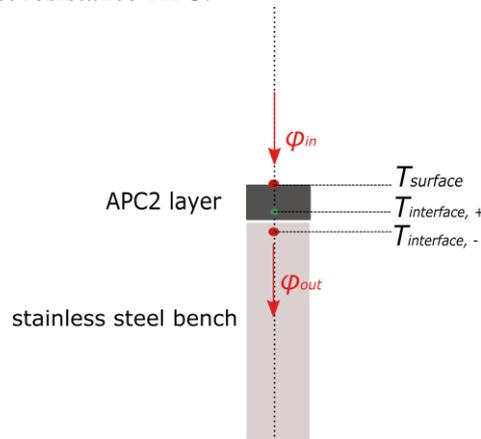


FIGURE 6: Schematic representation of the 1D Model for the calculation of Thermal Contact Resistances (TCR)

3 RESULTS AND DISCUSSION

After a complete calibration of the measurement devices implemented in the instrumented bench, the measurement performances of the bench were tested in terms of repeatability and scattering of results. The following section presents the results obtained for Configuration n°1 when the laser is in quasi-normal incidence ($\theta = 85^\circ$).

3.1 REPEATABILITY

The repeatability capability of the system was tested by irradiating a single composite layer 10 times in the exact same conditions: 10% of power (corresponding to 9 MW/m^2) during 25 ms. This latter was selected so that the maximum temperature reached by the layer remains under the melting point of the polymer. The shots were separated by one minute in order to ensure the cooling of the material. The surface temperature profiles were recorded with the infrared camera. Figure 7 shows the results for shot 1, 5 and 10 taken at 25 ms, that is to say, when the maximum temperature is reached. The temperature difference between the maximum values is around 4°C for an average of 270°C what comes down to a relative scattering around 1.5 %. Figure 8 shows the temperature profile recorded by the thermocouples TC1, TC2 and TC3 over the different tests. Again the profiles superpose in each cases proving the accuracy and robustness of the implemented metrology. For comparison purpose, the evolution of the temperature of spot center is displayed in dark red color according to the right axis. The difference between the two axis scales is of one order of magnitude and shows the ability of the system to measure accurately a wide range of temperature.

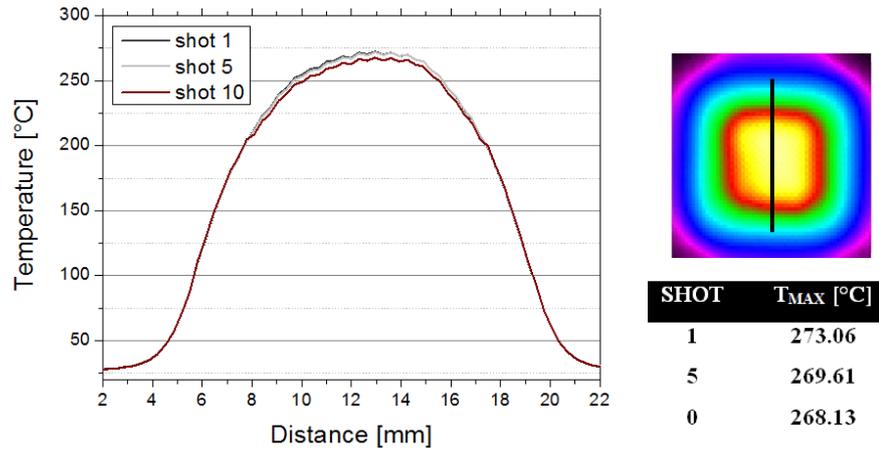


FIGURE 7: Evolution of the lateral temperature profiles taken at shots 1, 5 and 10.

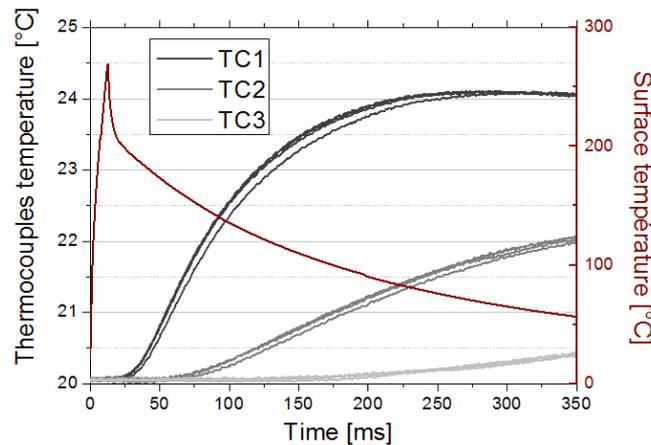


FIGURE 8: Evolution of the temperature in thermocouples TC1, TC2 and TC3 on the left axis; Evolution of the center surface temperature on the tape upper face during the 10th shot on the right axis (dark red).

3.2 DATA SCATTERING

The reproducibility of the results was then evaluated by testing different samples in the same conditions. Again the configuration was chosen so the maximum temperature was under the melting point of the matrix. 10 samples were illuminated during 50 ms with 10 % of the laser power. The thermal contact resistances (TCR) were calculated using the method presented in 2.3. Figure 9 shows the results obtained versus time. Firstly, it can be seen that the obtained TCR values extend over several order of magnitudes. The scattering of these experimental results is then large and can be attributed to a lack of homogeneity in the impregnation of the tape. Moreover, for all trials the thermal contact resistance increases during the heating step; for trials 1 and 3, the TCR is infinite at the start of the heating, even reaching infinite value for trials 1 and 4 at the very first time steps. This behavior can be explained by a lifting of the tape during the heating due to thermo-mechanical effects. Indeed, the gradient in the layer is high, from 340°C to room temperature, during 50 ms. After 50 ms, all TCRs came down to constant levels. The value of these levels depends on the quality of the contact after the lifting of the tape. The highest values, around 0.01 m².K/W, mean the contact is poor. On the contrary, lower values around 1.1e⁻⁴ m².K/W or even 1e⁻³ m².K/W are of relevant order of magnitude for a composite /metal contact.

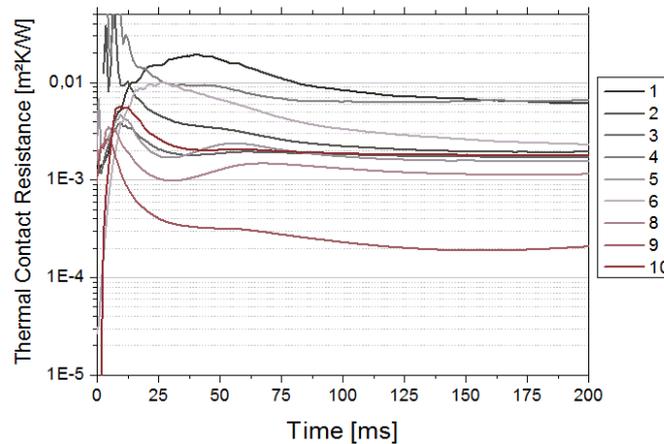


FIGURE 9: Thermal contact resistances (TCR) evolution versus time for 10 samples heated during 50 ms at 10 % of the laser output

Further developments are currently in progress to avoid the deformation of the composite tape during the heating and to improve the contact afterwards. The objective of this ongoing work is to measure the evolution of the thermal contact resistances during the heating and especially during the melting of the matrix.

4 CONCLUSIONS

An experimental bench has been developed to study the heat transfer occurring during the automated placement of carbon fiber reinforced thermoplastic composites. The final bench consists in two parts that have for goals, respectively, to finely analyze the thermal behavior of a composite tape heated by a laser diode, and to reproduce its behavior with the processing conditions.

After complete calibration of the thermal sensors and of the laser output, the performances of the first configuration were presented. It appeared that the measurements are reproducible for one same sample. Due to the thermo mechanical deformation of the composite tapes and the lack of impregnation quality of the prepreg, the dispersion of the thermal contact resistances measured over a series of different samples was high. Nonetheless, providing adequate modifications in the holding system and the use of enhanced quality composite layers, the capabilities of the system are promising. The heat transfer in the materials as well as the thermal contact resistances between the stainless steel bench and one composite ply and between several plies will be measured. The subsequent influence of different laser inclinations (0 to 10° incidence) and power supply levels (up to 2 kW) will be tested.

Finally, in the second configuration, thanks to this fine thermal metrology and the use of appropriate inverse methods, we will be able to assess the heat fluxes on each side before contact, at the interface during contact, but also the thermal contact resistance (TCR) evolution at this interface. The influence of different laser parameters and mold temperatures will also be investigated. Eventually, the collected data will be compared with a finite element 2D model of the whole process implemented on Comsol Multiphysics.

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REFERENCES

- [1] D. H.-J. a. Lukaszewicz, C. Ward, and K. D. Potter. The engineering aspects of automated prepreg layup: History, present and future, *Composites. Part B Engineering*, 43 (3), 2012, pp. 997–1009.
- [2] Z. August, G. Ostander, J. Michasiow, and D. Hauber. Recent developments in AFP for

- thermoplastic composites, *SAMPE Journal.*, **50** (2), 2014, pp. 30–37.
- [3] M. B. Gruber, M. a Lamontia, and B. J. Waibel. Automated fabrication processes for large composite aerospace structures: a trade study” *International. SAMPE Symposium Exhibit, 2*, 2001, pp. 1987–1997, 2001.
- [4] R. Pitchumani, J. W. Gillespie, and M. . Lamontia. Design and Optimization of a thermoplastic Tow-Placement Process with in-situ Consolidation, *Journal of Composite Material*, **31** (3), 1997, pp. 244–275, 1997.
- [5] M. A. Khan and R. Schledjewski, “Influencing factors for an online consolidating thermoplastic tape placement process, *Proceedings of the 17th international conference on composite materials*, Edinburgh, United Kingdom, 27–31, July 2009, Paper 4, pp 1-10.
- [6] C. M. Stokes-Griffin and P. Compston, “The effect of processing temperature and placement rate on the short beam strength of carbon fiber–PEEK manufactured using a laser tape placement process,” *Compos. Part A Appl. Sci. Manuf.*, vol. 78, pp. 274–283, 2015.
- [7] C. Ageorges, L. Ye, and M. Hou. Advances in fusion bonding techniques for joining thermoplastic matrix composites: A review, *Composites Part A Applied Science and Manufacturing*, **32**, 2001, pp. 839–857.
- [8] P. de Gennes, Reptation of a Polymer Chain in the Presence of Fixed Obstacles, *Journal of Chemical Physics*, **55** (2), 1971, pp. 572–579.
- [9] A. C. Loos and P. H. Dara, “Processing of Thermoplastic Composites,” Blacksburg, 1987.
- [10] J. K. Kim, Effect of cooling rate on interphase properties of carbon fiber / PEEK composites Part 1 . Crystallinity and interface adhesion,” *Composites - Part A Applied Science and Manufacturing*, **31**, 1999, pp. 517–530.
- [11] F. O. Sonmez and H. T. Hahn, Analysis of Process-Induced Residual Stresses in Tape Placement, *Journal of Thermoplastic Composite Materials*, **15** (6), 2002, pp. 525–544.
- [12] J. Tierney and J. W. Gillespie, Modeling of Heat Transfer and Void Dynamics for the Thermoplastic Composite Tow-Placement Process,” *Journal of Composite Materials*, **37** (9), 2003, pp. 1745–1768.
- [13] P. Patel, T. R. Hull, R. W. McCabe, D. Flath, J. Grasmeyer, and M. Percy, Mechanism of thermal decomposition of poly(ether ether ketone) (PEEK) from a review of decomposition studies, *Polymer Degradation and Stability*, **95** (5), 2010, pp. 709–718.
- [14] C. M. Stokes-Griffin and P. Compston. A combined optical-thermal model for near-infrared laser heating of thermoplastic composites in an automated tape placement process, *Composites Part A Applied Science and Manufacturing*, **75**, 2014, pp.104–115.
- [15] C. Nicodeau, “Modélisation du soudage en continu de composites à matrice thermoplastique,” Ecole Nationale Supérieure d’ Arts et Métiers, 2005.
- [16] W. Groupe, “Weld strength of laser-assisted tape placed thermoplastic composites,” Universiteit Twente, 2012.
- [17] R. Lichtinger, P. Hörmann, D. Stelzl, and R. Hinterhölzl, The effects of heat input on adjacent paths during Automated Fiber Placement,” *Composites Part A Applied Science and Manufacturing*, **68**, 2015, pp. 387–397.
- [18] C. M. Stokes-Griffin and P. Compston, “Investigation of sub-melt temperature bonding of carbon-fiber/PEEK in an automated laser tape placement process,” *Composites Part A Applied Science and Manufacturing* **84**, 2014, pp. 16–19.
- [19] A. J. Comer, D. Ray, W. O. Obande, D. Jones, J. Lyons, I. Rosca, R. M. O’ Higgins, and M. a. McCarthy, “Mechanical characterisation of carbon fiber–PEEK manufactured by laser-assisted automated-tape-placement and autoclave,” *Composites Part A Applied Science and Manufacturing*, **69**, 2015, pp. 10–20.
- [20] A. Barasinski, “Modélisations du procédé de placement de fiber” Ecole Centrale Nantes, 2013.
- [21] Y. Quilliet, S., Le Bot, P., Delaunay, D., & Jarny, Heat transfer at the polymer-metal interface - A method of analysis and its application to injection molding, *Proceedings of the 32nd National Heat Transfer Conference, Baltimore, MD (US), August 8-12, 1997*, pp. 11–13.
- [22] J. V Beck and B. Blackwell. Comparison of some inverse heat conduction methods using experimental data, **39** (17), 1996, pp. 3649–3657.
- [23] C. M. Stokes-Griffin and P. Compston, “Optical characterisation and modelling for oblique

near-infrared laser heating of carbon fiber reinforced thermoplastic composites,” *Optical Laser Engineering*, **72**, 2015, pp. 1–11, 2015.