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IDENTIFICATION OF THE THERMOPHYSICAL PROPERTIES OF POLYMER AND COMPOSITE MATERIALS DURING THEIR TRANSFORMATION

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ABSTRACT

Controlling the quality of industrial products requires an accurate comprehension of the material behavior during the several transformation phases. The exact modeling of the heat transfers taking place is crucial for manipulating rigorously the manufacturing processes. Though, this modeling necessitates the knowledge and the conform characterization of the material thermophysical properties which are the thermal conductivity, the specific heat and the specific volume. Currently, these properties are well defined in the solid state, however they are less mastered in the liquid state (for thermoplastics) and during transformation. The aim of this paper is to establish an inverse heat transfer method for predicting the variation of the thermophysical properties during the polymer transformation. This phase change corresponds mainly to the crystallization of the molten polymer to form a solid with an ordered internal arrangement of molecular chains. The concept of the inverse method consists in minimizing the difference between the numerical outputs obtained by a developed finite difference model and the temperature evolution of the material during its transformation. A hybrid optimization algorithm combining a stochastic algorithm with a deterministic one is adopted and its robustness is verified by using synthetic noisy data generated by the numerical model. A sensitivity analysis is conducted in order to test the feasibility of the parameters simultaneous identification. The expected results will allow to describe the variation of the thermophysical properties function of temperature T and the relative crystallinity α .

KEYWORDS: Thermophysical properties, Thermoplastics, Crystallization, Inverse method, Optimization

1. INTRODUCTION

Identifying the thermophysical properties of polymer and composite materials has become an important matter and holds significant importance in the several industrial applications. They contribute in several aspects such as the control and the validation of the manufacturing processes, analyses of the thermal and/or mechanical stresses and shocks, thermal fracture prevention, evaluation of manufactured material quality. The crystallinity of thermoplastics has a major impact on its thermal behavior. Indeed, the thermophysical properties are not only dependent on the temperature but also on the solidified fraction. The conventional characterization methods have been widely used to investigate the variation of these properties in the amorphous state and in the semi-crystalline state function of temperature [1] [2] [3] [4]. Previous experimental characterization have been conducted in the purpose of evaluating the change of thermophysical properties function of temperature and the crystallized fraction [5] [6]. Discrete methods were employed to estimate the properties of the semi-crystalline polymers in their final state with a fixed percentage of crystallinity and not during their crystallization. Moreover, some authors modeled the effect

of the crystallization on the thermal conductivity with the aid of models describing the conduction through heterogeneous media like composites. The fiber ratio is substituted by the relative crystallinity and the spherulites are assimilated to spherical crystallites embedded in amorphous matrix. The classical models are the Maxwell's model [7], Rayleigh's model [8] and Springer's model [9]. Le Goff *et al.* [10] compared the output of these models with the mixing rule and observed a negligible discrepancy due to the weak contrast between the thermal conductivity of the amorphous and the semi-crystalline phases.

Several apparatuses have been introduced to estimate the variation of the thermoplastics thermal conductivity function of temperature. The thermal response of the material is measured and implemented in an inverse model in order to identify the unknown parameters. These studies consisted in optimizing a set of the thermal conductivity values at predefined temperature levels. In this case, the thermal conductivity dependence of temperature was described by a piece-wise linear function. Le Goff *et al.* [11] recorded the evolution of the temperature at the centre of an injected polypropylene. An inverse method was then applied to compute the thermal conductivity of the polymer in its solid and liquid state while excluding the crystallization phase. Tardif *et al.* [12] estimated the thermal conductivity of a polypropylene by solving an inverse heat conduction problem. The identification was realized based on the temperatures evolution recorded within the material while being cooled from a temperature of 210 °C to the ambient at a cooling rate of 1 K/min. The thermal conductivities were then estimated at different temperature levels outside the crystallization phase. The results were consistent with the ones found by Le Bot [13], Le Goff [10] and with a hot guarded plate.

The traditional inverse heat conduction problems aimed to optimize the unknown thermophysical properties of the material in its final state [14] [15] [16] [17]. Therefore, these estimation methods consisted in identifying a fixed value of each property and not an evolutionary profile. The originality of this study resides in the fact that no previous variation rule is imposed to solve the inverse problem. The developed model will focus on optimizing the unknown parameters while taking into account the temperature variation and the transformation degree. A numerical model describing the temperature evolution coupled with the crystallization is developed and the reliability of the estimation technique is validated

2. NUMERICAL MODEL

2.1 Description and resolution of the numerical model A numerical resolution of the thermal problem was considered for the inverse method. The numerical model corresponds to the cooling of an injected thermoplastic within a mould of thickness $e=4$ mm. The one dimensional heat equation modelling the thermal response of the polymer is coupled with the exothermal heat generation taking place during the crystallization phase. The heat conduction problem is described by equations 1 and 2:

$$\rho(\alpha, T)C_p(\alpha, T)\frac{\partial T}{\partial t} = \frac{\partial}{\partial x}\left(\lambda(\alpha, t)\frac{\partial T}{\partial x}\right) + \rho(\alpha, T)\Delta H\frac{\partial \alpha}{\partial t} \quad \forall x \in]0; e[\quad \forall t > 0 \quad (1)$$

$$\frac{\partial \alpha}{\partial t} = n \times K_{Nak}(T) \times (1 - \alpha)[-ln(1 - \alpha)]^{1-\frac{1}{n}} \quad \forall x \in [0; e] \quad \forall t > 0 \quad (2)$$

With ΔH being the crystallization enthalpy, $K_{Nak}(T)$ the Nakamura coefficient function of temperature and n the exponent of Avrami. The traditional differential form of Nakamura [18] [19] was simplified to an equation more natural to implement and to solve numerically. Levy [20] proved the robustness and the accuracy of writing the crystallization kinetic in the following form:

$$\frac{\partial \alpha}{\partial t} = K_{Nak}(T).G(\alpha) \quad \forall x \in [0; e] \quad \forall t > 0 \quad (3)$$

With $G(\alpha)$ a function computed over the interval $[0,1]$. A thermal contact resistance of $10^{-2} W/m^2.K$ is

considered between the mold and the polymer. This value is based on the previous studies done by Le Mouellie *et al.* [21] about the heat transfer taking place during overmoulding processes. The boundary conditions considering the imperfect contact are written as:

$$-\lambda(\alpha, T) \frac{\partial T}{\partial x} = -\frac{T - T_{mold}}{R_1} \text{ for } x = 0 \quad \forall t > 0 \quad (4)$$

$$-\lambda(\alpha, T) \frac{\partial T}{\partial x} = \frac{T - T_{mold}}{R_2} \text{ for } x = e \quad \forall t > 0 \quad (5)$$

The initial conditions of the thermal problem are:

$$T(x, t = 0) = 573.15 \text{ K} \quad \forall x \in [0; e] \quad (6)$$

$$\alpha(x, t = 0) = 0 \quad \forall x \in [0; e] \quad (7)$$

The heat transfer problem is modelled using the finite difference method. The Crank-Nicolson scheme was adopted to discretize the one dimensional heat problem. The equations ought to be solved numerically are the following:

$$T^{k+1} = [A^{k+1}]^{-1} \times (B^{k+1} \times T^k + b) \quad (8)$$

$$\alpha^{k+1} = \alpha^k + \Delta t \times K_{Nak}(T^{k+\frac{1}{2}}) \times G(\alpha^{k+\frac{1}{2}}) \quad (9)$$

With A and B being a tridiagonal matrices containing the conduction terms and b the crystallization and the boundary vector. Since the heat equation is coupled with the crystallization kinetic, an iterative calculation is executed to simulate the thermal behaviour of the polymer. Initially, the values at the time t^{k+1} is considered equal to the values at the time t^k . Then, the values of the temperature and the relative crystallinity at t^{k+1} are computed by solving equations 8 and 9. At each time step, the calculations are repeated until the temperature and the relative crystallinity difference between two successive iterations is less than 10^{-3} and 10^{-6} respectively. The thermal problem was solved using Thomas algorithm since the matrices A and B are tridiagonal [22].

2.2 Effect of the time and space discretization on the resolution of the numerical model The finite discretization of the conduction problem in space and time allows the apparition of the term $R = \frac{\Delta t}{\Delta x^2}$. The sensitivity of the numerical outputs with respect to the time and space discretization (not shown here for reasons of space) has shown that the optimal time step is $\Delta t = 0.1 \text{ s}$ and the optimal element length is $\Delta x = 0.02 \text{ mm}$. In order to guaranty the convergence of the developed numerical model, the inverse of the Fourier number $Fo^{-1} = \frac{\Delta x^2}{a \times \Delta t}$ is computed in the molten and in the solid state. The values are shown in Table 1:

Table 1 Values of the inverse Fourier number.

State	Molten (T=400 K; $\alpha = 0$)	Solid (T=293 K; $\alpha = 1$)
Fo^{-1}	0.0336	0.0512

The value of Fo^{-1} remains lower than 0.5 when passing from the molten to the solid state proving the aptitude of the numerical model to solve the coupled heat problem for the adopted discretized scheme.

2.3 Evaluation of the numerical model The numerical model consists in simulating the cooling phase of the Polyamide 66 after being injected at a temperature of 573.15 K in a steel mold at a temperature of 293 K. The crystallization kinetics of PA 66 was investigated by Faraj [23] as well as the values of the thermophysical properties. For the sake of validating the developed model, the traditional mixing law was employed to solve the heat transfer problem [24]. The thermal conductivity and the volumetric specific heat are described by equations 10 and 11, where “sc” and “a” refer to the semi-crystalline and amorphous phases respectively.

$$\lambda(T, \alpha) = \lambda_{sc} \times \alpha + \lambda_a \times (1 - \alpha) \quad (10)$$

$$\rho C_p(T, \alpha) = \rho C_{p_{sc}} \times \alpha + \rho C_{p_a} \times (1 - \alpha) \quad (11)$$

The resolution of the thermal problem will generate the temperature and the relative crystallinity evolution at different points of the polymer’s thickness. Since the boundary conditions at both extremities are similar and the polymer is isotropic, the thermal problem is symmetric. The results obtained at the points located between 0 and $\frac{e}{2}$ are represented in Figures 1 and 2.

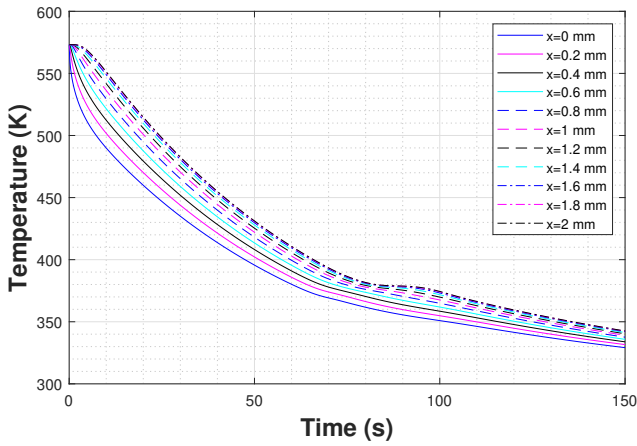


Fig. 1 Temperature evolution at different points of the polymer’s thickness

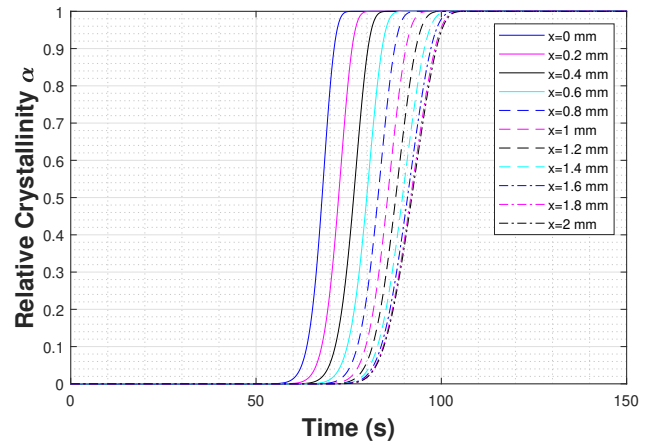


Fig. 2 Relative crystallinity evolution at different points of the polymer’s thickness

The temperature plateau detected in Figure 1 is due to the exothermal heat released during the crystallization of the polymer. It is noticed that the cooling rate is reduced as the thickness increases leading to a delay in the crystallization. Consequently, the crystallization occurrence is mostly significant at the center of the polymer.

In order to evaluate the developed numerical model, its results in pure conduction as well as the results of the finite element software "Comsol Multiphysics" were compared with an analytical model. A good agreement was obtained between the numerical and the analytical models and the difference in temperature is less than 0,1 K as shown in Figure 3. It is also noticed that the response of the numerical model is more exact than the one simulated by Comsol. The results of the numerical model in crystallization were then compared with Comsol Multiphysics. The backward Euler resolution method is used by the software and the coupling between the thermal equation and the crystallization is achieved through a specialized library built by Levy [25]. The same discretization in space and time is adopted for both numerical models. The differences in temperature and in crystallization kinetics are represented in Figures 4 and 5 respectively. It is observed that the temperature difference does not exceed 1,2 K while the difference in the crystallization kinetics reaches a maximum of 0,01. This temperature discrepancy is larger than the experimental measurement noise which

is around 0.025 K. Therefore, the outputs of the developed model are reliable to proceed with the inverse method.

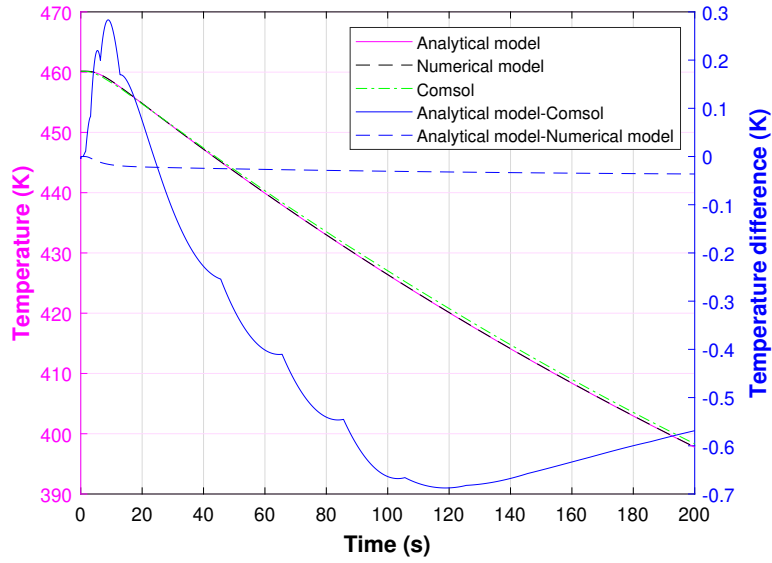


Fig. 3 Temperature evolution simulated by the numerical model, an analytical model and Comsol Multiphysics

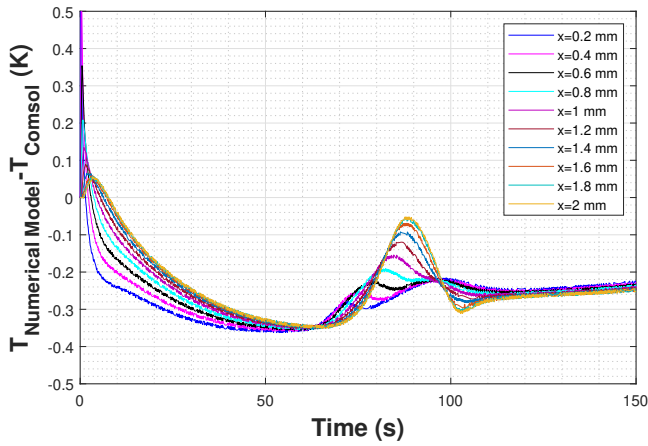


Fig. 4 Temperature difference between the numerical model and Comsol at different points of the polymer's thickness

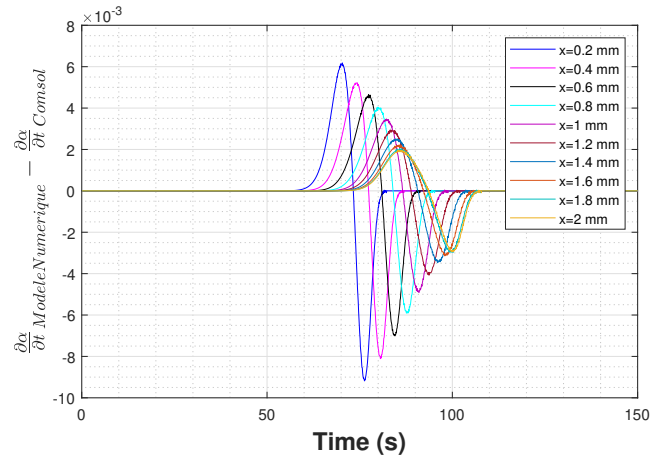


Fig. 5 Difference in crystallization kinetics between the numerical model and Comsol at different points of the polymer's thickness

2.4 Sensitivity to the thermophysical properties Prior to identifying the sought thermophysical properties, it is fundamental to observe the sensitivity of the model outputs with respect to the unknown parameters. This analysis is required to verify the feasibility of the estimation and to acknowledge for the possible existing correlations among the properties. Hence, the reduced sensitivity of the temperature with respect to the thermal conductivity, specific heat and density is computed. It is defined as:

$$S^*(\beta, t) = \frac{\partial Y(\beta, t)}{\partial \beta} \times \beta \quad (12)$$

With β being the parameter to identify and Y the output of the system.

The obtained results are plotted in Figure 6. The similar variation of the sensitivity curves during the overall exploitation time suggests that the thermophysical properties are correlated. In this case, the alteration of the model output can be related to any parameter of the thermal problem. Furthermore, the correlation matrix has been calculated as shown in equation 13. It appears more clearly that the thermal conductivity and the specific heat are highly correlated. This ascertainment may define the inverse problem as ill-posed preventing the simultaneous identification of the parameters [26]. Consequently, the study will be conducted towards identifying only the thermal conductivity of the polymer function of the temperature and the relative crystallinity.

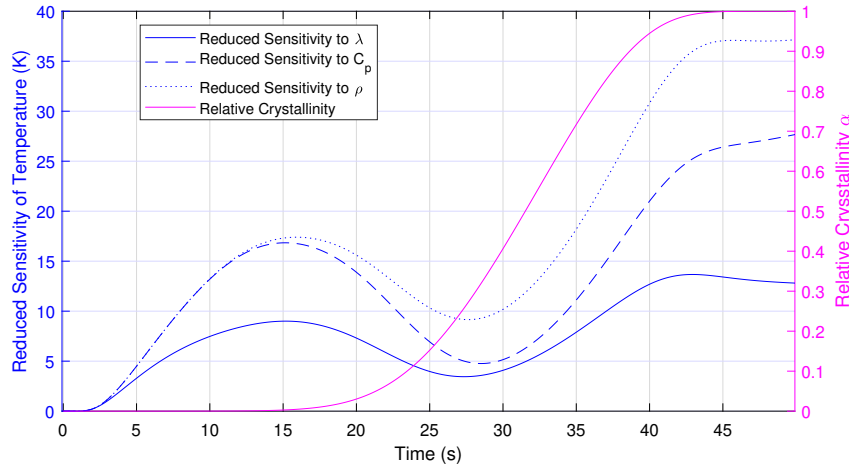


Fig. 6 Reduced sensitivity of the temperature with respect to the thermophysical properties.

$$\begin{pmatrix} \lambda & \rho & C_p & \\ 1 & -0.1883 & 0.9609 & \lambda \\ & 1 & -0.4458 & \rho \\ & & 1 & C_p \end{pmatrix} \quad (13)$$

3. INVERSE HEAT CONDUCTION PROBLEM

3.1 Description of the inverse problem For the current inverse method, optimization is the ultimate goal for the estimation of the thermophysical properties. The resolution of the inverse problem relies on minimizing an objective function representing the discrepancy between the experimental/synthetic results and the numerical results. The concept of the inverse problem is presented in Figure 7 along with a schematic representation of the measurement points within the injected polymer. The optimization algorithm keeps on adjusting the values of the thermophysical properties until the cost function satisfies a predefined criterion.

The cost function is described by the following formula:

$$J = \sum_{i=1}^n \int_0^{t_f} (T_{i,Experimental}(\beta) - T_{i,Numerical}(\beta))^2 dt \quad (14)$$

With n being the number of points where the temperature variation is measured/computed, t_f the experiment time length and β the set of thermophysical properties to identify.

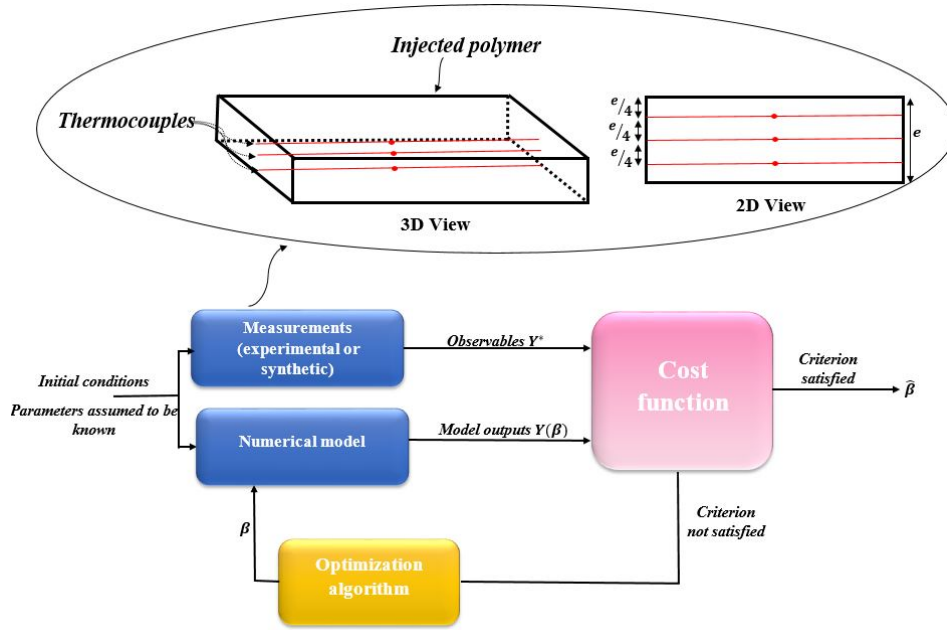


Fig. 7 Algorithm of the inverse problem.

3.2 Unknowns of the thermal problem One of the outputs of the inverse problem is a $m \times n$ matrix containing the thermal conductivity values corresponding to n predefined temperature levels and m possible values of the relative crystallinity (ranging between 0 and 1). It is defined as follows.

$$\begin{pmatrix} \lambda_{11} & \lambda_{12} & \dots & \dots & \dots & \dots & \dots & \lambda_{1n} \\ \lambda_{21} & \lambda_{22} & \dots & \dots & \dots & \dots & \lambda_{2n-1} & \lambda_{2n} \\ \vdots & \vdots & \vdots & \ddots & \ddots & \ddots & \vdots & \vdots \\ \lambda_{m1} & \lambda_{m2} & \dots & \dots & \dots & \dots & \lambda_{mn-1} & \lambda_{mn} \end{pmatrix} \quad (15)$$

This matrix will assist in computing the thermal conductivity of the polymer during its crystallization. It is found by performing a double linear interpolation function of the temperature and the relative crystallinity. However, the polymer in the molten and in the solid states has a fixed relative crystallinity ($\alpha = 0$ and $\alpha = 1$ respectively). Therefore, in these states the thermal conductivity is only dependent of the temperature. The conductivity of the molten polymer is defined as a set of values corresponding to the possible temperature levels. Similarly, a different set is assigned for the thermal conductivity of the solid polymer function of temperature.

3.3 Optimization algorithm The identification of the thermal conductivity values can be achieved by applying a stochastic optimization algorithm or a deterministic one. Since the actual problem is non-linear and presents a relatively high number of parameters, the use of a stochastic approach is required to avoid getting trapped in a local minimum. Among the stochastic approaches, the metaheuristic techniques are nature inspired and zero-order algorithms. In the current study, the aptitude of the Particle Swarm Optimization algorithm (PSO) [27] and the Genetic Algorithm (GA) [28] [29] for identifying the unknown parameters is tested. The stochastic algorithm (PSO or GA) is combined with a deterministic method. The final estimation values optimized by the stochastic technique will be implemented as initial values to the gradient based algorithm. The interior point algorithm able to solve non-linear convex problems, is adopted [30].

3.4 Estimation results The temperature profiles simulated by the developed numerical model are implemented in the inverse method. The estimation technique is selected by comparing the results given by the particle swarm optimization algorithm and the genetic algorithm, both coupled with a deterministic method that takes place at the end of the stochastic algorithm. The numerical model simulates the cooling of the injected PA 66 from a temperature of 400 K to a temperature of 345 K. According to the numerical results, the crystallization of the considered polymer occurs between 365 K and 385 K. In this manner, the thermal conductivity is identified in the molten state ($T > 385 K$ and $\alpha = 0$), during the crystallization phase and in the solid state ($T < 365 K$ and $\alpha = 1$). The temperature difference between the unknown values of the thermal conductivity is 10 K and the difference in relative crystallinity is 0.1.

The objective function corresponds to the temperature differences at three distinct points of the polymer's thickness. Since the crystallization is mostly detected at the center of the polymer (Figure 1), the three measurement points are considered at the center of the polymer and at both sides at a distance of $e/4$ from the edges. The identified matrix is compared with the original one calculated with the help of the mixing rule (equation 10) and used to produce the synthetic results. The relative error of each identified value is computed in order to compare the accuracy of the algorithms.

Without noise The results computed without synthetic noise are presented in Figures 8 and 9. They are recapitulated in Table 2 to highlight the maximum relative difference between the estimated results and the actual values of the thermal conductivity. It is noticed that the results obtained using the Genetic Algorithm are more accurate than the values found by the Particle Swarm Optimization algorithm.

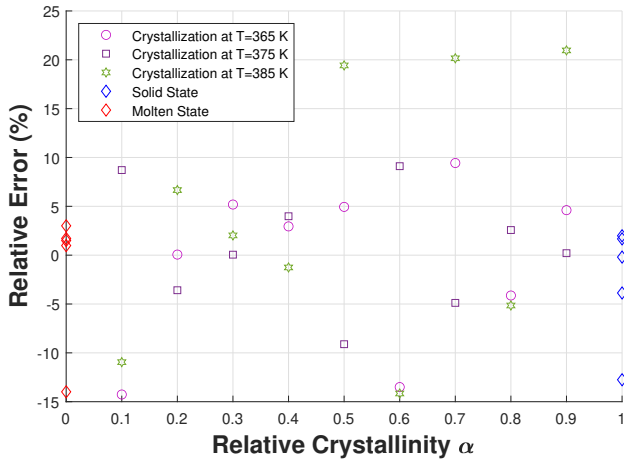


Fig. 8 Relative error of the estimated thermal conductivity using PSO without noise

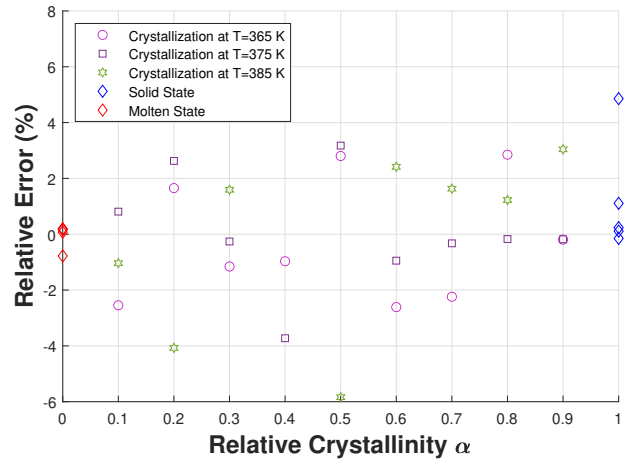


Fig. 9 Relative error of the estimated thermal conductivity using GA without noise

Table 2 Maximum absolute relative difference between the estimated values and the actual values of the thermal conductivity.

State	PSO	GA
Solid	3.004%	1.107%
During crystallization	20.15%	6.41%
Molten	3.87%	0.196%

With noise Several level of noises are added to the numerical results. Since the GA has proved to be more effective than PSO, the identification is done using the GA coupled with the interior point algorithm. The

relative errors of the obtained results are shown in Figures 10, 11, 12 and 13. The good agreement between the estimated and the actual values of the thermal conductivities, even for relatively important level of noises, verify the feasibility and the accuracy of the estimation strategy. Furthermore, the identified values of the thermal conductivities with a noise of $0.025\text{ }^{\circ}\text{C}$ are more precise than the ones estimated with a noise of $0.01\text{ }^{\circ}\text{C}$. Effectively, the added noise of intensity $0.025\text{ }^{\circ}\text{C}$ is generated by a Gaussian distribution based on the experimental measurements while the other level of noises are created arbitrarily. It is also noted that the stochastic aspect of the calculations may lead to unsimilar results in each of the presented cases.

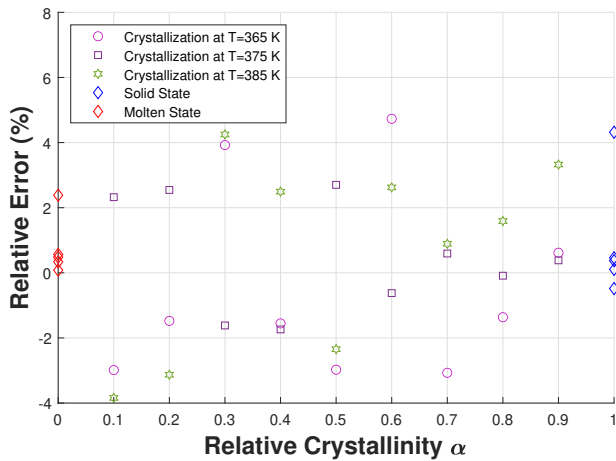


Fig. 10 Relative error of the estimated thermal conductivity using GA with a noise of $0.01\text{ }^{\circ}\text{C}$

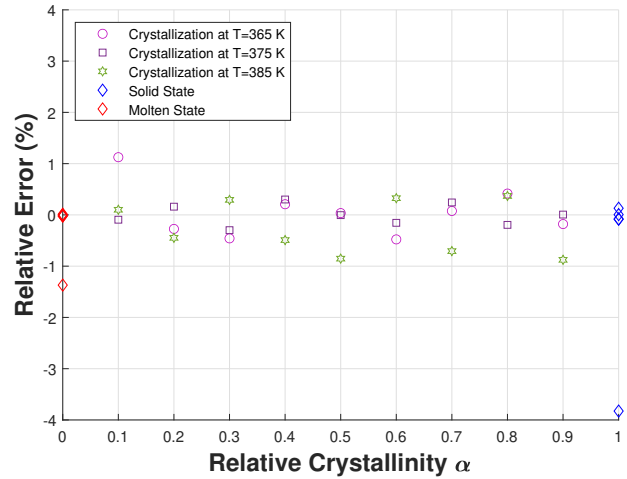


Fig. 11 Relative error of the estimated thermal conductivity using GA with a noise of $0.025\text{ }^{\circ}\text{C}$

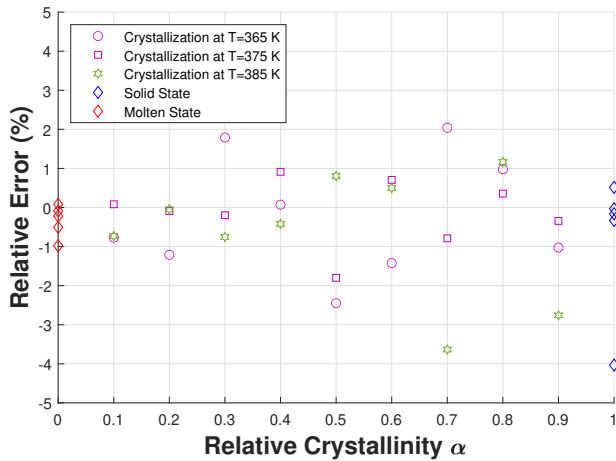


Fig. 12 Relative error of the estimated thermal conductivity using GA with a noise of $0.1\text{ }^{\circ}\text{C}$

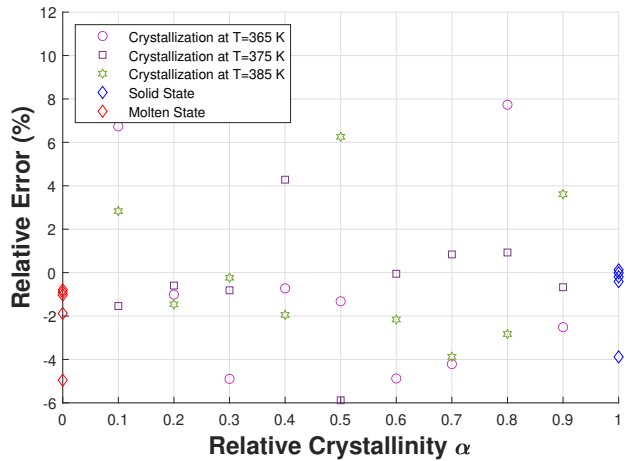


Fig. 13 Relative error of the estimated thermal conductivity using GA with a noise of $0.3\text{ }^{\circ}\text{C}$

4. CONCLUSION

An estimation technique of the thermal conductivity function of the temperature and the relative crystallinity has been investigated in this paper. For this purpose, a numerical model simulating the crystallization of thermoplastics is established and compared to an analytical model and the software Comsol Multiphysics.

The performances of the Particle Swarm Optimization algorithm and the Genetic Algorithm were compared and it was found that the genetic algorithm is more compatible with the present thermal problem. This work constitutes a preliminary step for the experimental identification. Further analysis will be made to investigate the sensitivity of the thermal problem with respect to the number of increments in temperature and crystallization. This study will assist in determining the number of identifiable unknowns based on their correlations. The apparatus developed by Le Mouellic *et al.* [21] will be used to measure the temperature evolution at different points of an injected polymer during its cooling phase. Therefore, three K-type thermocouples of 125 μm diameter are stretched in the moulding cavity. These measurements will be implemented in the developed identification technique to optimize the thermal conductivity. The identified values will contribute in visualizing the actual evolutionary profile of the thermal conductivity during the crystallization phase. An experimental campaign aiming to measure the thermal response of the polymer is under execution to finalize the inverse method. The next step will consist in applying the innovative inverse method on a composite material in order to identify the thermal conductivity variation during crystallization.

NOMENCLATURE

λ	Thermal conductivity	$[W/(m.K)]$	α	Relative crystallinity	$[\]$
C_p	Specific heat	$[J/(kg.K)]$	$ Fo$	Fourrier number	$[\]$
ρ	Density	$[kg/m^3]$	x	Thickness	$[mm]$
T	Temperature	$[K]$	n	Avrami exponent	$[\]$
R	Thermal resistance of contact	$[m^2.K/W]$	K_{Nak}	Nakamura coefficient	$[s^{-n}]$

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