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To design the cure process of thick composite parts: experimental and numerical results

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To design the cure process of thick composite parts: experimental and numerical results

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The cure degree must be as uniform as possible during the manufacture of polymer matrix composite components in order to have a product without defects. For thick composite components, this condition is not often respected; in fact, the cure degree trend between the core and the external surface is different causing structural and geometrical/dimensional unconformities. In most cases, these problems can be solved through a redesign of cure process in terms of thermal cycle, in fact that one recommended by furnisher is usually suitable for thin components. The optimization of cure thermal cycle should include several performance criteria for the production system such as the targeted cure degree, the targeted maximum temperature of the part and the duration of the cure cycle, as well as the production system limitations such as the maximum allowable heating rate, the maximum allowable cooling rate and so on. A previous work shows a method to optimize the cure degree of a thick composite component. The present work presents an indirect way to validate the proposed method: some experimental tests have been carried out by implementing the cure cycle identified by this method, the values of temperatures have been recorded by thermocouples and the obtained temperature trend has been compared with that due to numerical approach. Further considerations on the cure degree and the cure rate have been deduced. The experimental results show a good agreement with the numerical ones.

Keywords: thick composite components; cure process; cure degree; cure rate; optimization method

Introduction

One of the critical processes during the manufacture of polymer matrix composite component is the cure one; in fact during cure some defects may arise, such as resin degradation and residual stress. These conditions may happen especially in thick composite components; in fact the high thickness causes a series of phenomena that could bring to low degree of cure, to exothermic temperature peak or other problems. The thermal conductivity of the resin is so low that the heat reaching the core is not enough for curing the matrix; this involves low mechanical properties of the component.[1] Moreover, the heat developed from curing reaction is not enough dispersed in order to avoid an increase of the temperature and a deterioration of the resin.[2] These phenomena are the more important as more thickness is elevated.

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The increase of temperature in the inner zones of thick composite components speeds up the cure, so the trend of the cure degree between the core and the external surface is different, in fact the resin in the inner zones cures before than that of the outer ones. Due to chemical shrinkage of polymerization, this phenomenon lets internal stresses arise during the cure process. In order to reduce the effects of this inhomogeneity, it is necessary to design opportunely the thermal process to obtain the intersection cure degree trend (α_{cross}) between the core and the external surface close by after gel point (AGP). The AGP plays a significant role on the magnitude of process-induced residual stresses: the cure degree along the thickness of components must be uniform to reduce curing stresses.

Residual stress is critical for thick or large parts. The residual stresses can give rise to component cracking and distortion [3,6]; this causes problems in assembly that lead to increased costs. For parts made with asymmetric laminates or dissimilar materials, residual stress is caused by stratification itself, while for symmetric parts residual stress is generated by uneven curing and solidifications.[7] It has been demonstrated that in the case of thick composite laminates, internal stresses during the curing stage can reach a peak generally connected to a faster cure of the core. Further stresses can arise during the cure due to differential strains between the part and tooling on which it is manufactured, whereas aluminium or steel tools have a much higher expansion coefficients than composite parts: they tend to stretch the parts as they heat up and small shear stresses can arise at the tool interface causing tension in the part.[1,2,8–16] AGP also plays a significant role on the magnitude of the process-induced residual stresses, as demonstrated in the works,[14–16] so cure degree must be uniform in thickness of components to reduce curing stresses. However, for thick composite components, this condition is not often respected because the cure degree trend between the core and the external surface is different. In order to reduce the effects of this inhomogeneity, it is necessary to design opportunely the thermal process to obtain the intersection cure degree trend (α_{cross}) between the core and the external surface close by AGP. It is very important to remember that the cure cycle recommended by prepreg manufacturer is suitable only for thin laminates, while it causes the aforementioned problems during the cure of the thicker ones.[18]

So, the quality of composite components is largely affected by the cure cycle that must be designed and optimized: the common problem in the cure cycle design of a thermoset composite material manufacturing is how to set-up the time-temperature-pressure profile in such a way that certain criteria are fulfiled and the result is optimal.[18–28] A cure thermal cycle should comply with several targets, such as the minimum cure degree, the maximum temperature inside the laminate and the unevenness of the cure degree trend; the cure cycle optimization should satisfy these targets and optimize other goals, such as the minimum required time.[29] The literature offers a trial and error procedure to optimize the cycle, but it is expensive and time wasting; its results are acceptable, but not always optimal. The first improvement to this kind of procedure is developing numerical model to simulate the phenomena that happen during the process: this procedure is less expensive than the previous, however a lot of simulations have to be run to achieve the optimal cure cycle. In a previous work [30], a new method for cure cycle optimization, that specifies how to modify the cycle in order to gain a part without defects with only few simulations, was presented.

The present work shows an indirect way to validate the method presented in the previous paper. The new method has been applied on a high thickness laminate through a numerical approach and an optimal cure cycle has been achieved. The obtained cure cycle has been implemented by some experimental test during which the trend of the temperature has been recorded by thermocouples. The experimental trend has been compared with the numerical one.

Mathematical models for cure cycle

Finite element (FE) models, that analyse the thermo-chemical properties of the component, are typically used to determine the temperature and the degree of cure inside the part.

To analyse the complete phenomenon, the entire model is generally divided into four sub-models: thermochemical, flow, void and stress.[7] However, among the aforesaid sub-models, only thermochemical one allows to define the temperature trend in the whole system, which consists of component and mould. This trend is generated by some thermal fluxes that are driven from the outside or that are generated inside by the reaction, and other parameters depend on it, such as viscosity and degree of cure, in fact polymerization reaction is controlled by heat. So, in order to simulate the cure process of a composite material component it must be taken into account the energy balance on the system, that is:

$$\rho_c \, c_{p,c} \, \frac{\partial T}{\partial t} = \nabla (k_c \, \nabla \, T) + \rho_r \, V_r \, \dot{Q} \tag{1}$$

In which ρ represents material density, c_p is the specific heat, t is the time, ∇ is the Laplace operator and k is the thermal conductivity coefficient of the composite material, that is anisotropic, $\dot{Q}$ is the heat generation rate by chemical reaction, V is the volumetric percentage and T is the temperature. The subscript c and r refer, respectively, to composite and matrix properties, while f is for fibre reinforcement. In this work, composite material is considered as a homogeneous one, in fact the physical properties are considered The thermal conductivity along fibres direction (longitudinal) is calculated by a weighted average on volumetric fraction, while that one along the direction normal to the fibres (transversal) is

$$\frac{k_t}{k_r} = 1 - 2\sqrt{\frac{V_f}{\pi}} + \frac{1}{B}\left[\pi - \frac{4}{\sqrt{1-C}} \tan^{-1} \frac{\sqrt{1-C}}{1 + B\sqrt{V_f/\pi}}\right] \tag{2}$$

The coefficients B and C that appear in the previous relation are expressed as:

$$B = 2\left(\frac{K_r}{K_f} - 1\right) \tag{3}$$

$$C = B^2 \frac{V_f}{\pi} \tag{4}$$

In some case, the transversal thermal conductivity is obtainable by Nielsen equations [30,31]:

$$\frac{k_c}{k_r} = \frac{1 + ABV_f}{1 - B\psi V_f}, \quad A = k_E - 1, \quad B = \frac{(k_f/k_r) - 1}{(k_f/k_r) + A}, \quad \psi = 1 + \left(\frac{1 - V_{\max}}{V_{\max}^2}\right) V_f \tag{5}$$

where k_E is the generalized Einstein coefficient (1.84), $V_{\max}$ is the volume fraction of maximum fibres with recommended value 0.82 [32].

Composite density can be derived by the volumetric fraction of fibres and matrix [32,33]:

$$\rho_c = \rho_r V_r + \rho_f V_f \tag{6}$$

while specific heat can be computed as:

$$c_{p,c} = (V_r \rho_r c_{p,r} + V_f \rho_f c_{p,f})/\rho_c \tag{7}$$

in which subscript 'r' refers to resin, 'f' to fibres and c to composite. Fibres mass fraction can be got by:

$$m_f = \frac{\rho_f V_f}{\rho_f V_f + \rho_r V_r} \tag{8}$$

while the resin one is the one's complement.

In order to determinate the heat generation rate, it must be considered that it depends on the total heat of reaction H_r and on the cure rate ($\mathrm{d}\alpha/\mathrm{d}t$) [34]:

$$\dot{Q} = \left(\frac{\mathrm{d}\alpha}{\mathrm{d}t}\right) H_r \tag{9}$$

And the cure rate can be expressed as [8,35]:

$$\frac{\mathrm{d}\alpha}{\mathrm{d}t} = K(T) \cdot f(\alpha) \tag{10}$$

where $f(\alpha)$ can be calculated in many ways, depending on reaction model, that are listed below [35]:

- First-order relation

$$f(\alpha) = 1 - \alpha \tag{11}$$

- *N*th order relation

$$f(\alpha) = (1 - \alpha)^n \tag{12}$$

- *N*th order relation with autocatalysis

$$f(\alpha) = (1 - \alpha)^n (1 + K_2 \alpha) \tag{13}$$

- Prout–Tompkins relation

$$f(\alpha) = \alpha^m (1 - \alpha)^n \tag{14}$$

in which n and m are reaction orders, while K_2 is a constant, and they can be determined through laboratory tests, such as differential scanning calorimetry (DSC).[36] The exponential coefficients m and n are determined according to ASTM 2070. The function $K(T)$ in Equation (10) can be determined with an Arrhenius relation:

$$K = A_c \exp\left(-\frac{E}{RT}\right) \tag{15}$$

in which E is activation energy, R is the universal gas constant and A_c is frequency factor.

Once assigned equation governs what happens in the domain, it is necessary to assign boundary conditions on surface: it could be a fixed temperature or a heat flux.

Validation of the new methodology to cure thick composite components

The new methodology proposed in the previous work is based upon the observation of the trend of the cure degree in the core (A) and in the surface (C) of a composite during the first thermal cycle (I TC), see Figure 1: initially the cure rate of the surface is higher than that of the core, so the cure degree of the surface is higher too. Later, the two rates become equal (point $\dot{\alpha}^X$ in Figure 1(b)) and then the cure rate of the core overcomes that of the surface, so the cure degree of the core exceeds that one of the surface (point α_{cross} in Figure 1(a)); however, the α_{cross} value is high and far from AGP value. The new proposed methodology introduces a second dwell in $\dot{\alpha}^X$ in order to move the intersection point α_{cross} closer to AGP. As denoted in Figure 2, with this new dwell, the cure rate in A and C has a reverse trend. In C the reaction slackens immediately, while in A it continues for a certain time, due to thermal inertia, and then is slowed down: in this way the convergence of the cure degree between A and C is obtained with a lower value of α_{cross}, and the temperature is maintained constant till the two cure degree trend intersect. The length of this new dwell is about 10 minutes, so its influence on total cycle length is minimal.

The procedure followed in this work is shown in Figure 3. The starting point is a typical FEM model, with its CAD model, mesh, material properties, boundary and initial conditions. The new methodology generates a cure cycle that means it identifies the boundary conditions, i.e. mould temperature and thermal insulation, and the model parameters that are the initial conditions of the simulation. Once the simulation has been carried out, it is verified that the reached maximum temperature is below the

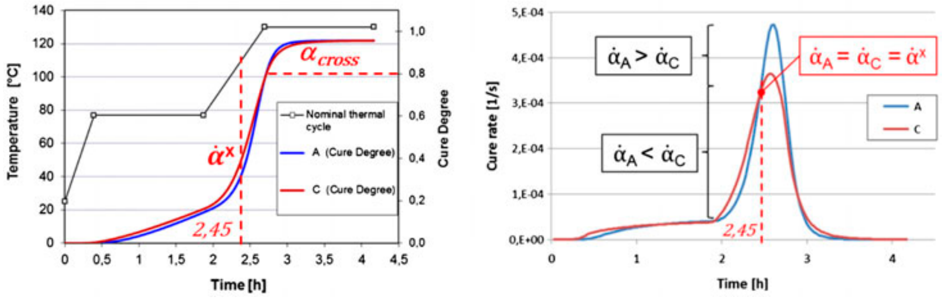

Figure 1. Typical trend of cure degree and cure rate for a high thickness composite component.

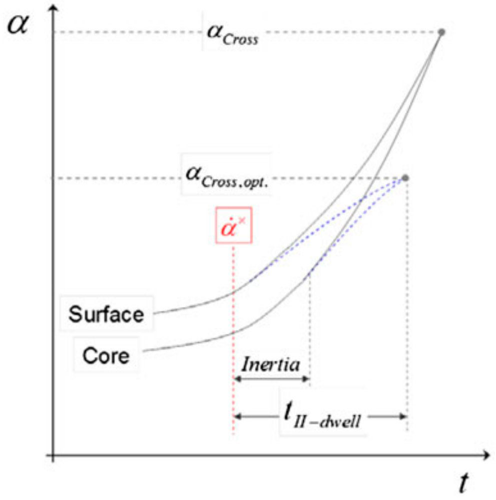

Figure 2. Effect of second dwell on cure degree.

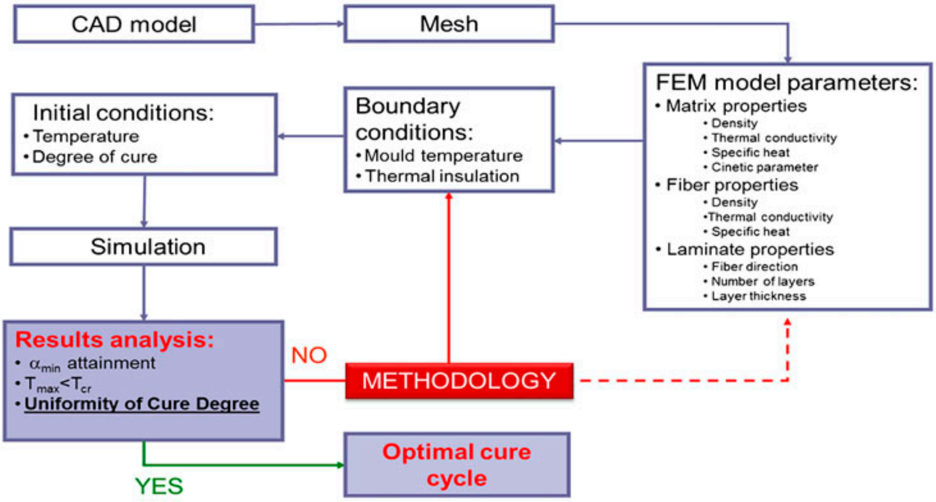

Figure 3. Procedure to design an optimized cure cycle.

critical one and the degree of cure is uniform and above the minimum value. If these conditions are satisfied, the designed cycle is an optimal one and the procedure is stopped; on the contrary, the new methodology must be applied again to define a new thermal cycle and so on. The optimal cure cycle has been used to carry out some experimental tests and the obtained results have been compared with the numerical ones.

The considered real component has been a plate 100 mm × 100 mm with a thickness of 20 mm. It has been made up of 66 prepreg plies. As aforementioned, the AGP condition is a key factor of cure cycle, so a critical material for this condition has been chosen: the cure degree of the gel condition is very low (28%), and other composite material data, determined through experimental test, are in Table 1. The component is constituted by prepreg of carbon fabric and epoxy resin. Above-mentioned material has a cure temperature between 70 and 100 °C and starts to degrade at 120 °C.

FE modelling of cure process

As regards FE modelling of cure process, the heat exchange equation considered is (1), in which the heat generation is calculated according to Prout–Tompkins relation (14). Physical characteristics of this composite material, such as thermal conductivity, density and specific heat, are calculated by the physical characteristic of fibres and matrix by relations (2), (6) and (7), in which the volume fibres fraction considered is $V_f = 0.60$. The values of thermal and kinetic material properties are reported in Table 1.

In order to reduce the computation time, some consideration can be done about the model, as reported in Figure 4. First of all, due to load and geometric symmetry, only a quarter of the model has been modelled. Moreover, after a previous simulation, it has been found out that the edge effect was irrelevant and so only a slice of the mould-prepreg assembly could be simulated. Therefore, a 2D model has been developed. As regards boundary conditions, a condition of symmetry is applied in the centre. Moreover, a temperature is applied on the upper and lower surface (obviously the temperature is variable with time, according to the established thermal cycle) and a condition of insulation is applied on the side surface.

Table 1. Parameters of composite material used for experiments (carbon fibres and epoxy resin).

Physical property	Value
ρ (kg/m^3)	1584
K_t (W/(mK))	0.59
H_r (J/g)	100
c_p (J/(kgK))	931.6
A_c (1/s)	$8.99 \cdot 10^{10}$
K_1 (W/(mK))	6
E (kJ/mol)	91.45
m	0.3
n	1.3

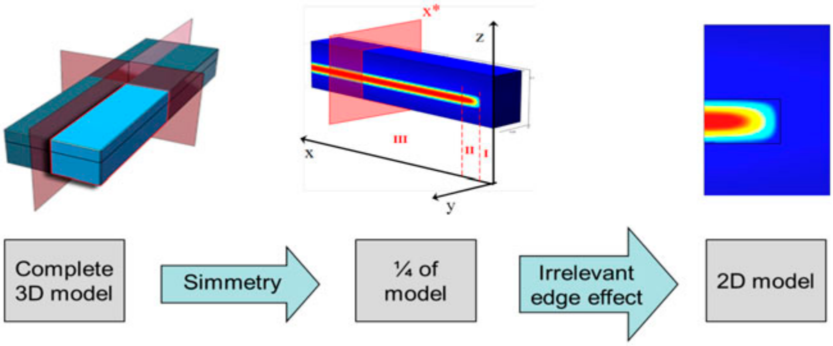

Figure 4. Numerical model simplifications.

The early thermal cycle applied is taken from prepreg datasheet. It schedules only one heat rise and one dwell: the former goes from room temperature to 373 K at 2 °C/min and then the latter lasts for 100 min at this temperature. This thermal cycle is denoted in Figure 5.

Numerical results

The cure process for this laminate has been simulated with PAM-RTM®, and the results are reported in Figure 6: it can be seen that the temperature in C (in the surface zone) follows closely to that one imposed by the thermal cycle, so the conditions on maximum temperature and on final cure degree are respected. Moreover, these conditions are respected in the centre zone (A) too, even if a little deviation can be noticed at the end of heat rise. However, observing the obtained cure degree trend in Figure 7, it is possible to see that the same value of cure degree in A and C (that is the α_{cross} condition) is reached when the cure degree is equal to 0.54, i.e. very far from AGP (equal to 0.28). Therefore, it has been necessary to apply the new methodology developed in the previous work in order to redesign the thermal cycle.

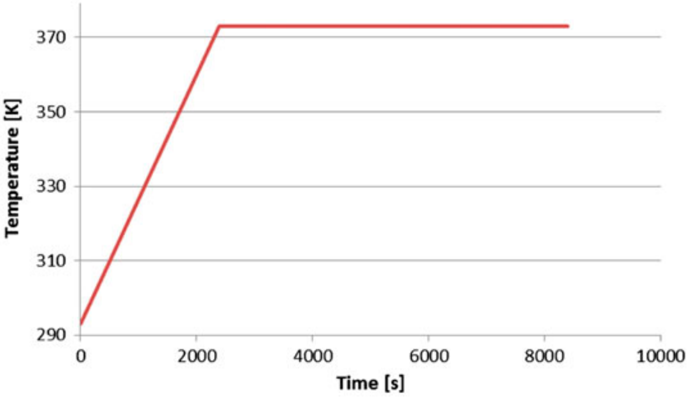

Figure 5. Early cure cycle.

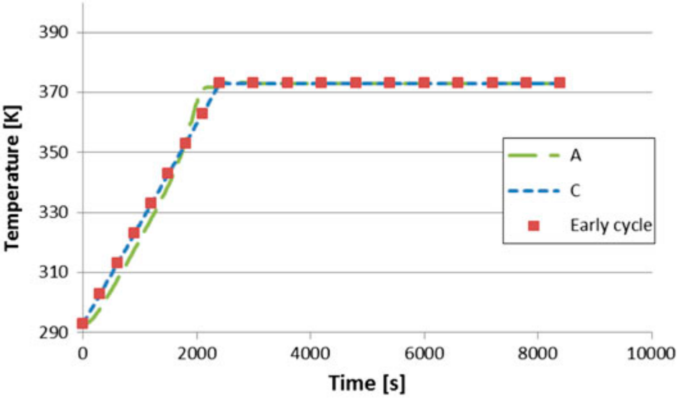

Figure 6. Temperature trend from early cure cycle.

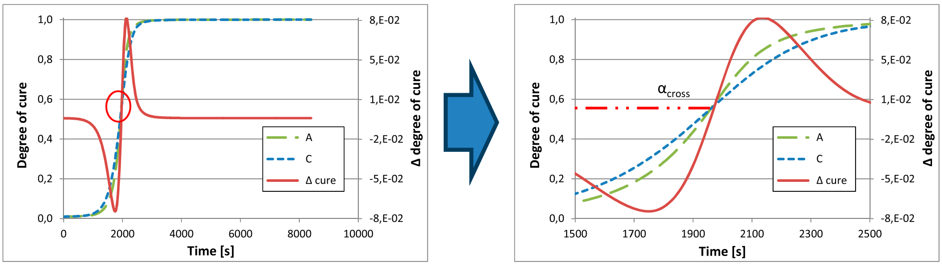

Figure 7. Trend of cure degree from early cure cycle.

According to the methodology, a second temperature dwell has been added in thermal cycle applied $\dot{\alpha}^x$, which occurs after 1750s, see Figure 8. In order to keep unvaried the cycle duration, this second dwell has lasted only 10 min, so the new temperature cycle is illustrated in Figure 9.

As expected with this new thermal cycle (Figure 10), the condition on maximum temperature is respected, in fact there is only a little thermal peak on the new first dwell, but it does not reach the maximum allowed temperature. As regards degree of cure trend, it can be seen from Figure 11, α_{cross} is 34% that is closer to AGP than the previous value. Therefore, even this aim is achieved.

Experimental results

As shown previously, the main factor affecting the cure process is the temperature. Therefore, if the temperature trend inside the real component is equal to that from the numerical results, it is assumed that the cure trend to be the same.

The composite component has been polymerized by a low-cost closed mould technology, whose diagram is shown in Figure 12. Five samples have been manufactured. The moulds have been constituted by two plates made of 5083 aluminium, 400 mm × 400 mm with a thickness of 10 mm, in order to guarantee a good stiffness and a uniform heat distribution. The heating system has been made up of six flexible heaters, constituted by a wire-wound element, that have been positioned directly in contact with the aluminium plates, and all together provides 1800 W heat power, which is sufficient to ensure a heating cycle as defined in the simulation step. The heaters have been controlled through a PID regulator, a Watlow 'Ez-Zone PM', which allows to

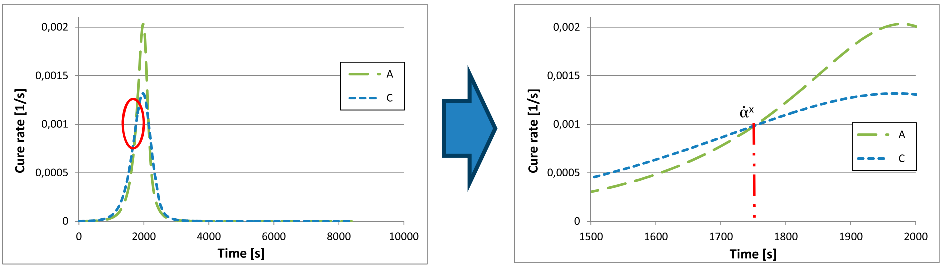

Figure 8. Trend of cure rate from early cure cycle.

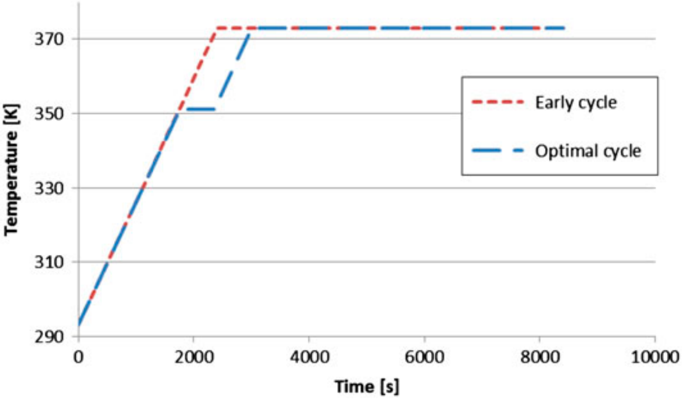

Figure 9. Optimal cure cycle.

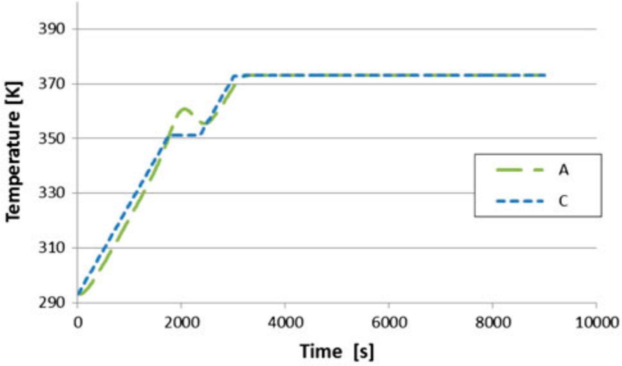

Figure 10. Temperature trend from optimal cure cycle.

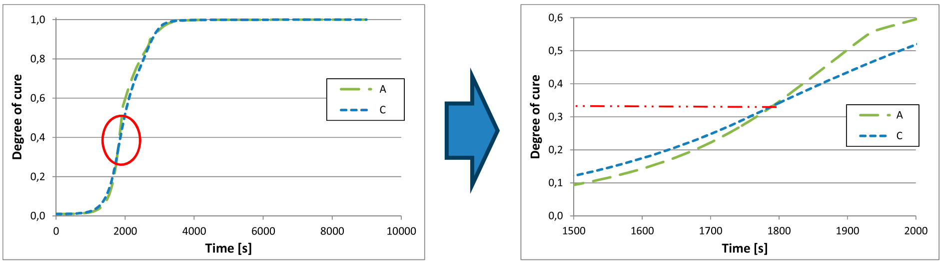

Figure 11. Trend of cure degree from optimal cure cycle.

control carefully the temperature of the system, thanks to a 15A ‘no arc’ relay. The closing system has been constituted by a vacuum bag that wraps the whole equipment and supplies a constant compaction force during the whole cure cycle. The two plates have been coated with a spray release agent, to facilitate the demoulding after cure,

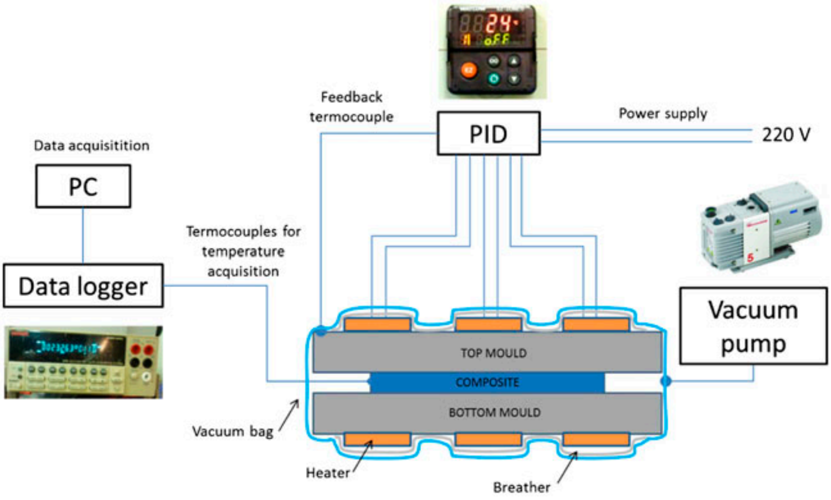

Figure 12. Diagram of equipments used for experimental tests.

then 66 prepreg sheets have been cut to size and layered on the lower mould, for a total thickness of 20 mm, and some thermocouples have been inserted for monitoring the temperature trend during the cure cycle, as shown in Figure 13. Finally, the moulds have been covered with the heater, wrapped in the breather film and closed in the vacuum bag (Figure 14): the whole system has been ready and the cure cycle could start, while the temperature field has been recorded by a computer.

The validation is based on the comparison between experimental and numerical temperature trends inside the component. The component has been cured according to the cycle developed by the previous described procedure (see Section 3), the temperature inside and on the surface has been recorded during the cure and it has been compared with that due to numerical approach. Both early and optimal cycles have been checked.

The average values of the temperature trend, which has been recorded during the early cure cycle, are shown in Figure 15. The standard deviation of the data has been equal to 6% of the average value and, therefore, it has been neglected. All the considered curves are referred to average values. The temperature trend discovered in real component for the early cure cycle is very close to that due to numerical approach, in

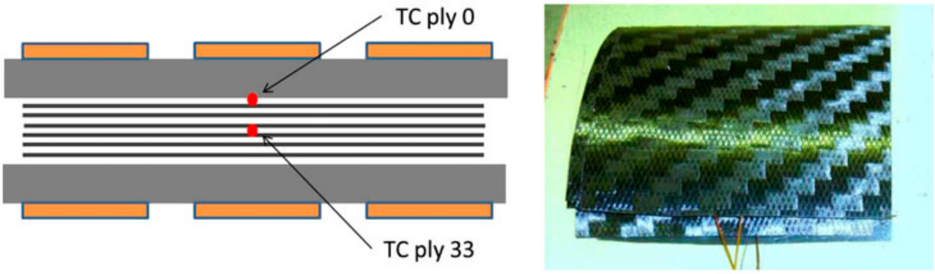

Figure 13. Thermocouples' position during cure process.

Figure 14. Experimental cure equipment.

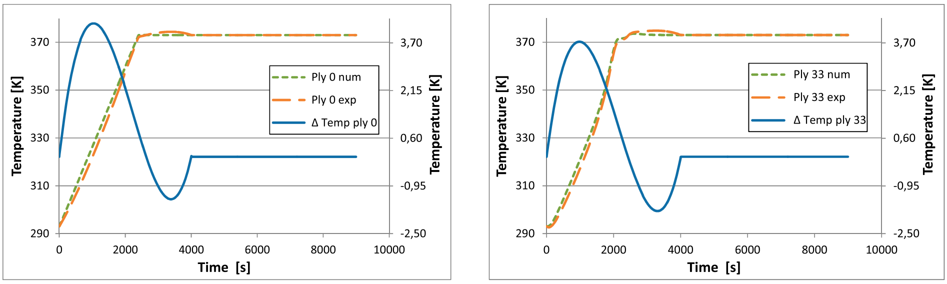

Figure 15. Comparison of temperatures' trend for early cure cycle.

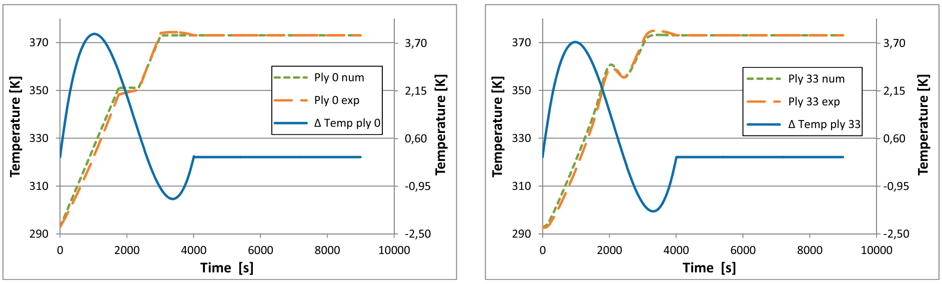

Figure 16. Comparison of temperatures' trend for optimal cure cycle.

fact the maximum temperature difference between numerical and experimental result is 4 K for the surface and 3.7 K for centre. Moreover, this shifting can be found only in the first part of the cycle, during the heating. The same considerations can be done also for optimal cycle, in fact from Figure 16 it can be seen that the maximum temperature difference is 3.7 K for centre and 3.8 K for the surface. So, the temperature trends matched very well and the methodology is validated.

Conclusions

The present work shows the validation of a new methodology to design thermal cycle for thick components manufactured by a closed die technology. In fact, in this case the trend of the cure degree can be different between core and surface by involving residual stresses, cracking and distortions that cause problems during assembly with an increase of the manufacturing costs.

In order to reduce the effects of this difference, it is necessary to design opportunely the thermal process in order to obtain the intersection cure degree trend (α_{cross}) between the core and the external surface close to AGP, which plays a significant role on the magnitude of process-induced residual stresses.

To validate experimentally the methodology, a high thickness component has been cured according to different cycles, and the temperature has been recorded during the cure and it has been compared with that from numerical simulations. If the temperatures are close, also the cure degree should be close. It has been found that the obtained temperature trend is very close to that from numerical results, since the difference is less than 4 K.

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