Effect of energy director thickness on thermal diffusion and joint quality during ultrasonic welding of CF/PEEK composites

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Abstract. Thermoplastic composites offer new possibilities for the aerospace industry. Large and thin structural parts can be processed rapidly and more cost-effectively than thermoset composites since the latter need to undergo lengthy curing reactions. An additional advantage is the possibility of being assembled by welding processes. However, joining thermoplastic composites remains challenging due to the lack of knowledge of the impact of process parameters (time, pressure, temperature) on the welded joints. A polymer film named energy director (ED) is usually set up at the interface to enhance the weld resistance. A combined experimental-numerical approach was carried out to estimate the influence of ED thickness on the thermal diffusion within the assemblies and the mechanical resistance of welds in the ultrasonic welding (USW) process. The studied materials are unidirectional carbon fibre-reinforced polyetheretherketone (CF/PEEK) and 50 µm and 250 µm polyetherimide (PEI) films as ED. Thermocouples (TCs) are inserted into the PEI layer to measure the temperature evolution during USW. Besides, the thermal profile is obtained based on the simulation of conduction phenomena through the thickness of the material using the finite element method (FEM). The effect of the ED through-thickness model points out the evolution of the temperature profile with time during the USW process for different PEI thicknesses. For both ED thicknesses, the temperature reached is above the PEI glass transition (220 °C) allowing macromolecular interdiffusion. According to our results, only the first ply reaches the melting temperature of PEEK, showing that the composite parts are not thermally affected during USW. Because of the high conductivity of unidirectional CF/PEEK composites, a thinner ED leads to a too-short heating time at the interface. Associated with too high power in a short time, a lower weld resistance of APC-2 parts is obtained by single lap shear for 50 µm PEI layer, 12 MPa, compared to 43 MPa obtained for 250 µm. A higher PEI thickness at the interface slows down the cooling step, resulting in a deeper interdiffusion of PEI and so, a stronger weld.

Introduction

Ultrasonic welding is a process that makes it possible to obtain good-quality welds quickly and affordably [1,2]. In USW, the heat is generated at the interface by friction between the two parts through high-frequency vibrations. Indeed, the process can be easily implemented at an industrial scale and the cycle times are in the order of seconds. Ultrasonic welding does not need any specific material property [3]. No additional device is necessary to generate heat in the welding area compared to induction and resistance welding for which, susceptor and resistor are required, respectively. The development of an industrially viable solution for ultrasonic welding of CF/PEEK would give this family of composite materials an undeniable advantage over thermosetting composites.

Many parameters affect the ultrasonic welding quality, such as welding time, pressure, amplitude and type of energy director [4,5]. Monitoring the thermal diffusion is crucial to relate process parameters to welds' performance. Nevertheless, measuring the temperature during welding processes is challenging and the works related are rare [3]. Non-invasive techniques do not exist yet to assess the temperature profile at the interface during welding. Due to the closed contact between the two composite parts with an integrated PEI layer, it is difficult to measure the temperature and almost impossible to control it. Moreover, the very short cycle times during USW reduce the means available to measure the temperature. Any device placed between the two specimens would alter friction phenomena, thus, changing heat generation [6,7]. Besides, some works report thermal profiles during USW obtained by simulation of thermal transfer [8,9], but none of them focuses on ED thickness.

In this work, the thermal analysis conducted on CF/PEEK composites using different thicknesses of PEI are displayed. The micro-thermocouples were chosen to minimize their influence during welding. The experimental section presents quasi-non-intrusive temperature measurements where temperature profiles are analyzed through the influence of USW parameters. The mechanical resistance of welding joints and process parameters for two PEI thicknesses on CF/PEEK are correlated to the thermal profiles. The thermal diffusion through the sample thickness from the interface is investigated from a FEM analysis in the numerical section.

Materials and Methods

Experimental methods.

Continuous carbon fibre-reinforced polyetheretherketone composite, named APC-2 from Cytec Industries and polyetherimide, named Ultem 1000 from Sabic, have been used.

The composite plates were produced by compression with Pinette Emidecau Industries Lab800 thermo-compression device. Two types of plates are manufactured: (1) APC-2 plates and (2) APC-2 with a 250 µm or 50 µm PEI layer on top plates. A stack of 14 unidirectional plies of APC-2 was made to obtain a mirror symmetry lay-up $[0/90]_7$. The PEI film was integrated on the APC-2 plates.

The thermocouples were 80 µm diameter K-type from Omega. Chromel and Alumel threads were supplied separately and welded using the air arc welding technique. TCs are inserted according to the procedure described by Bonmatin et al. [10]. The TCs were deposited and covered with a metal mass of 5 kg on the composite containing the PEI film to insert them into the polymer layer. The assembly was then placed in an oven for 40 min at 290 °C. Then, the TCs were extended to connect them to a Logger GL980 midi data recorder from Graphtec Corporation.

Omega 4X ultrasonic welder from Mecasonic was used for the experiments. It is welding equipment with a working frequency of 20 kHz and a maximum load of 3000 N. The displacement amplitude of the waves generated by the generator is amplified by a factor of 1.7 by the booster and by a factor of 1.7 by the sonotrode.

The quality of the assemblies was assessed by lap shear strength (LSS). The assemblies tested were made following ASTM D1002, with samples of 100 x 25 mm^2 corresponding to length and width respectively, and an overlapping area of 10 x 25 mm^2. The Single Lap Shear (SLS) tests were performed on the Instron Universal 33R4204 tensile machine equipped with a 100 kN load cell with a crosshead speed of 2800 N.min^{-1}. The value of LSS [MPa] was calculated with Eq. 1.

$$LSS = \frac{F_{max}}{l_0 \cdot w} \qquad (1)$$

In the SLS test, the strength of a weld depends on the load at the breaking point F_{max} [N], the length of the weld zone l_0 [mm] and the width w [mm].

Numerical methods.

To carry out the simulation, the FEM has been chosen. It consisted of the simplification of differential equations for numerical use. These methods, introduced for many years, are still widely used for the resolution of thermal problems [11,12]. The configuration applied for the simulation was a simple one-dimensional (1D) thermal conduction model in which the in-depth heat diffusion throughout the material part, especially in the first layers of composites was considered. Abaqus software from Dassault System was used for the numerical analysis of heat transfer. The model considered the thermal and physical properties of the polymer changing with temperature and melting heat. In this case, the heat source term was considered to be null because no volumetric internal energy was generated. A non-stationary thermal model was developed, where the software used the local heat equation (Eq. 2).

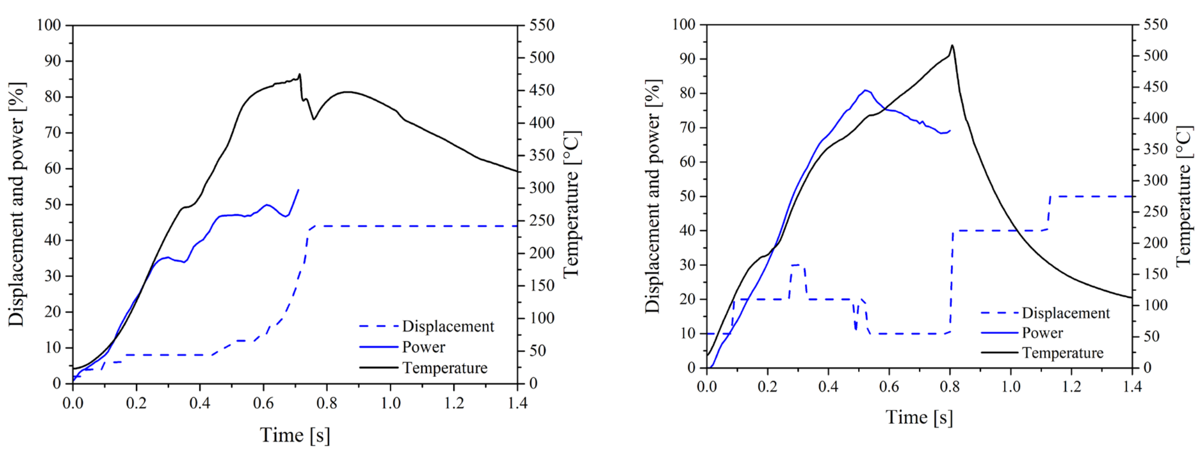
$$div\left(k\vec{\nabla}T\right) = \rho C_p \frac{\partial T}{\partial t} \tag{2}$$

where k is the thermal conductivity [W.(m.°C)$^{-1}$], T is the temperature [°C], ρ is the material density [kg.m^{-3}] and C_p is the heat capacity [J.(kg.°C)$^{-1}$]. The left-hand term corresponds to diffusion whereas the right-hand term corresponds to the transient heat state.

Results and Discussion

Experimental analysis.

During the welding process, the power [W] and displacement of sonotrode [mm] were recorded over time [s] by the machine, and they were plotted to identify different stages of USW according to Villegas's work. The welding machine can work in different modes, mostly the welding is controlled by time or displacement of the sonotrode. The time-controlled welding allows finding the optimum displacement of the sonotrode where the process is stopped in the stage before degradation of the material [13]. The temperature measurements were taken by thermocouple integrated into the PEI film during welding for every 1 µs. The three curves of power [%], displacement [%], and temperature [°C] are presented as a function of time [s] in Fig. 1. The maximum power of the machine considered for the calculation is 3000 W and the maximum displacement represents the thickness of two PEI layers, 0.5 mm and 0.1 mm.



(a) (b)

Fig. 1. Temperature monitoring with thermocouples during USW of APC-2 composites with integrated (a) 250 µm PEI for an amplitude of 68 µm, a load of 2.5 bar and a displacement of 30 % (b) 50 µm PEI for an amplitude of 68 µm, a load of 2.5 bar and a time of 0.8 s.

For the assembly of CF/PEEK with 250 µm PEI, the optimized displacement-controlled mode was applied at 30 %, Fig. 1.a, to melt the first plies of adherents. Regarding the welding of CF/PEEK with 50 µm PEI, the displacement-controlled mode at 40 % was not enough to weld the assembly while at 50 % high degradation of material was observed, so the time-controlled mode with optimized 0.8 s has been applied, Fig. 1.b. The same trend on the temperature curves during the welding step from room temperature to the maximum one can be observed. The latter reached 475 °C at 0.714 s and 517 °C at 0.807 s with a heating ramp towards 800 $K.s^{-1}$. Then, very rapid cooling is observed with a ramp towards 600 $K.s^{-1}$ and 1600 $K.s^{-1}$. ED with smaller thickness causes higher power of welding around 80 % with only two stages. At first, heating of the ED at the interface occurs due to the phenomenon of friction. The sonotrode does not move significantly because the materials involved in this step are still rigid and have not yet undergone a change of state. Then, the PEI continues to warm up. It begins to flow as a result of nucleation (viscoelastic heating mechanism controlled by surface friction heating) and the growth of hot spots at various places in the area of overlapped composite parts. The flowing of the energy director after reaching its glass transition causes a decrease in power and a displacement of the sonotrode. It can be assumed that the ED is not completely molten (lack of the third stage) and it seems that the first plies of composites do not reach the melting temperature of PEEK. So, there is no risk of degradation or delamination of PEEK composite parts during USW with these parameters. Displacement of the sonotrode until the end of welding achieves 50 %, which means that the thermocouples were in contact with the composites which explains the fast cooling rate. Due to high conductivity, carbon fibres contribute to very efficient heat diffusion.

After temperature monitoring, the mechanical strength was calculated. LSS are 43 ± 5 MPa and 12 ± 2 MPa for CF/PEEK with PEI of 250 µm and 50 µm, respectively.

Numerical conditions.

The transient thermal model was applied to the same welding configuration as the previous section, i.e. two layers of PEI, as seen in Fig. 2. The composite parts are 2 mm thick, and each PEI layer was 0.25 mm or 0.05 mm thick. A narrow transverse slice corresponding to the stacking of the three materials was designed in two dimensions (2D) by Abaqus. Therefore, a conduction model was performed, allowing to obtain the through-thickness thermal fields. To create this model, the following assumptions were used:

1) The interfaces between PEI and CF/PEEK were not perfect and thermal contact resistance was taken into account,
2) The studied materials were only PEI and CF/PEEK and the material properties varied with the concentration of each one, a mix of both materials was not considered,
3) Taking into account the thermal anisotropy of the composite, only the through-thickness properties of materials were considered,
4) The thermal behaviour of the sonotrode, steel plates or other tools was not considered,
5) Finally, only conduction was considered (neither convection nor radiation).

With these hypotheses, the conduction model gave an approximate thermal evolution at any point of the thickness and the evolution of temperature gradients over time.

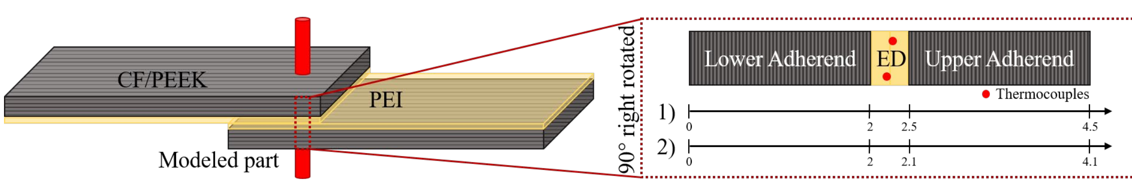


Fig. 2. Through-thickness model of the welding configuration, the distance through the thickness (mm).

The model is concentrated on the thermal properties of the CF/PEEK because such properties evolve much with temperature when PEEK changes from a glassy state to a flow state or a molten state. These properties are presented in Table 1. Those of PEI are presented in Table 2.

Table 1. Thermal properties of APC-2 [14].

Temperature [°C]	Thermal conductivity [W.(m.°C)$^{-1}$]	Specific heat [J.(kg.°C)$^{-1}$]	Density [kg.m^{-3}]
0	0.42	800	1601
50	0.52	930	1598
100	0.60	1040	1593
150	0.70	1260	1586
200	0.70	1300	1575
250	0.70	1400	1563
300	0.75	1550	1551
350	0.68	1650	1537
400	0.65	1700	1524

Table 2. Thermal properties of PEI [15].

Temperature [°C]	Thermal conductivity [W.(m.°C)$^{-1}$]	Specific heat [J.(kg.°C)$^{-1}$]	Density [kg.m^{-3}]
0 - 400	0.22	1248	1270

To consider the interface of CF/PEEK and PEI, the thermal conductance used for the model is presented in Table 3.

Table 3. Thermal conductance between CF/PEEK and PEI.

Temperature [°C]	Thermal conductance [W.(°C)$^{-1}$]
0 - 400	1000

Elements used for the model are four-node linear heat transfer quadrilateral elements, called DC2D4 in Abaqus software. The mesh is composed of two elements across the width of the studied area, as seen in Fig. 3. Two layers of 250 µm PEI have been meshed with 20 elements of 12.5 µm thick while for 50 µm PEI they have been meshed with 5 elements of 10 µm thick. The APC-2 layer is single bias meshed with an element size varying from 12.5 µm to 0.1 mm, and the mesh is increasingly finer near the PEI layer.

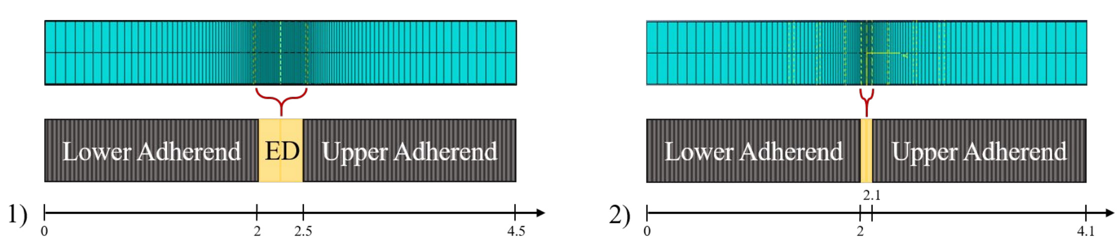


Fig. 3. Mesh elements of the out-of-plane model, the distance through the thickness (mm).

As recorded experimentally, the initial temperature of the specimen was considered to be at 23°C for 250 µm PEI and 20°C for 50 µm PEI. Therefore, this initial temperature was applied to the whole model. Adiabatic boundary conditions were used on the lateral sides, as well as on the upper and lower external surfaces of the composites.

The recorded temperature profile, shown in Fig. 1, was applied to the PEI layer presented in Fig. 3. At $t_0 = 0$ s, the temperature corresponds to the initial temperature. Then, the experimental temperature ramp was applied up to the value of 475°C at 0.714 s for 250 µm PEI and 517°C at 0.807 s for 50 µm PEI. This thermal profile was applied at the interface between the two integrated PEI layers.

Transient heat transfer analysis was used for this model and only the nodal temperature (NT) was computed as output data to understand the time and spatial evolution of the temperature field along the thickness of the assembly. This model contains two steps corresponding to the heating phase and the cooling phase. The heating phase lasts 0.714 s for 250 µm PEI and 0.807 s for 50 µm PEI as measured experimentally and the cooling phase lasts 15 s.

Simulation analysis.

Nodal temperatures were computed and their temporal evolution was compared, during the cooling phase, with the experimental thermogram (black line) recorded and plotted in Fig. 4. Three of the four curves correspond to different locations of the numerical model, showing respectively the interface between two integrated PEI layers (blue dashed line), the interface between APC-2 and the integrated PEI layer (green dashed line) and the interface between first and second composite ply (red dashed line).

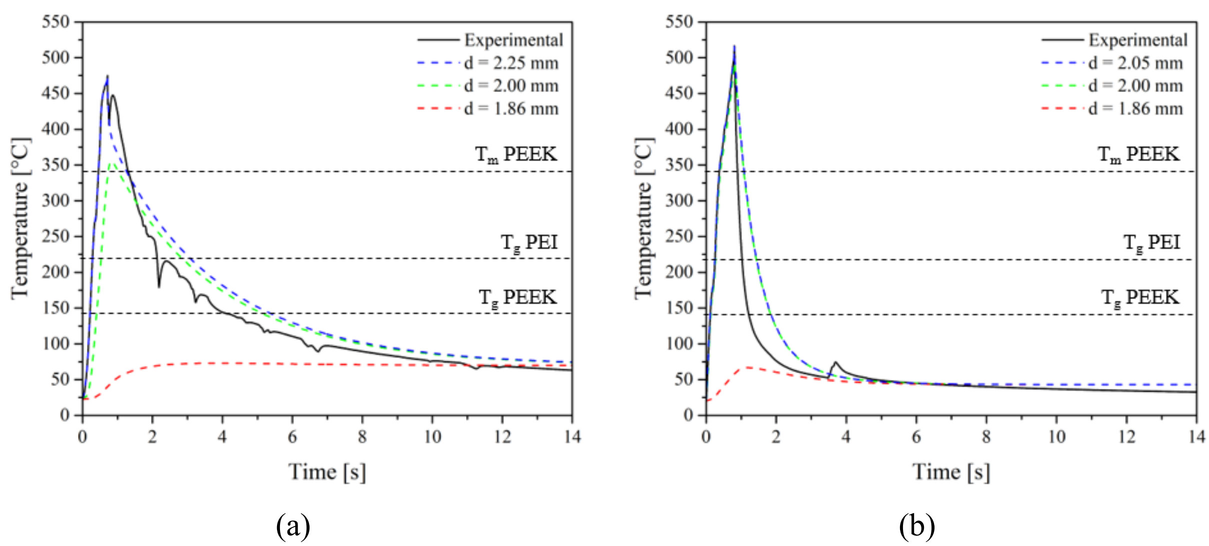


Fig. 4. Experimental and numerical thermograms during UW (a) for 250 µm PEI (b) for 50 µm PEI.

Comparing the experimental value to the numerical thermogram at the interface between the integrated PEI layers (d = 2.25 / 2.05 mm), where the TC are located, a slight difference can be highlighted. The cooling ramp is slower in the model, exhibiting either a too-high numerical contact conductance or a gradient of thermal properties in the interphase zone, which is not considered in the model and the numerical temperature starts to get closer to fit the experimental one. One hypothesis is that input material data from the literature do not fit the studied material. Moreover, the model does not take into account some heat exchanges like convection or exchanges with the jaws (adiabatic upper and lower surfaces boundary conditions). Regarding the material, the composite is made of unidirectional PEEK thermoplastic with carbon fibre. The thermal conductivity of CF/PEEK is higher than the one of the PEEK and so, when the heat moves to APC-2, it is mostly evacuated by the fibres. Moreover, in the case of CF/PEEK with 50 µm PEI, the TC significantly affects the diffusion of heat. The small thickness of ED and displacement during USW causes contact between TC and carbon fibre. The through-thickness modelisation also does not take into account transverse material properties and only propagates the heat through the thickness. Thus, every simulated temperature could be considered slightly overestimated.

Regarding the curve at the interface between APC-2 and the integrated PEI layer (d = 2.00 mm), the maximum reached temperature is 355°C for 250 µm PEI and 494°C for 50 µm PEI. The APC-2 composite reaches its melting (T_m) during welding in this configuration. It means that PEEK melting occurs within the first ply.

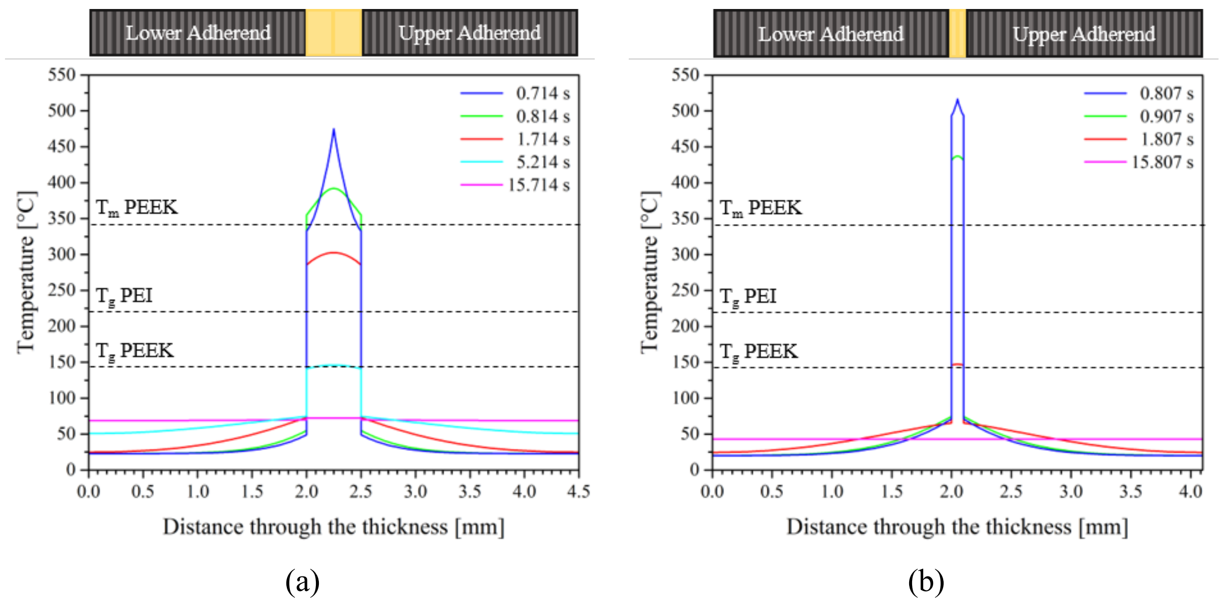


Fig. 5. Temperature profiles through the assembly right after the end of the vibration step with different welding times (a) for 250 µm PEI (b) for 50 µm PEI.

The spatial evolution of the temperature, right after the ultrasonic welding process and during the cooling step, is plotted along the thickness in Fig. 5. The true distance along the path with a reference value between the integrated PEI layers is plotted against the temperature. Different welding times are shown from t_c = 0.714 / 0.807 s (end of vibration step) to t = t_c + 15 s (end of the cooling step). It can be noticed that the profile is symmetrical from the middle of the integrated ED and, as expected, it is due to the symmetry of the stacking sequence of the model (geometry, material and section), loading and boundary conditions. Also, the curves present breaking points at a distance of 2.00 mm (PEI/PEEK interface), 2.25 /2.05 mm (PEI/PEI interface) and 2.50 / 2.10 mm (PEI/PEEK interface). As the thermal contact resistance was taken into account for the non-perfect interface between PEI and PEEK, a drastic drop in temperature is observed after 4.5 s for 250 µm PEI and 1.0 s and 50 µm PEI.

At the temperature t_c as the thermal loading is applied between two integrated PEI layers, the temperature in the middle of ED shows its peak at a value of 475 / 517°C. It is above the T_g of PEI, measured at 220°C, which implies that all the PEI is flowing. Moving to the interface between PEI and the composite layer, it appears that the temperature decreases to reach 355 / 494°C. This thermal evolution highlights the fact that ED, right after the ultrasonic loading, is partially flowing. The flowing material is located at the interface between PEI layers and allows interdiffusion

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between those two PEI layers, it is the condition to get a strong weld. Concerning composite parts, the maximum temperature reached during heating is above T_m of PEEK, measured at 340°C, which contributes to interdiffusion of the first plies. The time t_c is the starting point for the transfer of heat from the ED to the composite.

At $t = t_c + 0.1$ s, the heat flux has already started to be transferred from the two integrated PEI layers to CF/PEEK composites. The breaking point is no longer visible because the contact between integrated PEI layers is considered to be perfect. The ED temperature decreases and its maximum temperature is 392 / 437°C in the middle of the ED. The heat moves from the middle to the exterior, i.e. from the highest one to the lowest temperature. Regarding the composite layer, its maximum temperature is 355 / 435°C at the interface and still above its T_m giving mobility to the amorphous phase of the PEEK. At $t = t_c + 1$ s, in the case of thicker ED, the temperature is still above T_g of PEI while for the smaller thickness of ED, the temperature is already below. The same temperature for thicker ED is obtained after 5.214 s.

For the rest of the cooling process, the temperature at the centre of the ED decreases while it increases at the end of the assembly reaching the quasi-thermal equilibrium at the end of the cooling step, $t = t_c + 15$ s.

To conclude, in the studied configuration with a welding time of 714 / 807 ms, the temperature is above the T_g of PEI in almost the entire thickness of ED. This indicates that interdiffusion is possible between the PEI layers. Concerning APC-2, the maximum temperature reached is 355 / 494°C. It can be assumed that the PEEK matrix is thermally affected during welding allowing interdiffusion in the first ply, the second ply remains not affected. However, in the case of 50 µm PEI, the time to create a strong weld between ED and composite was too short causing low mechanical resistance. LSS results agree with the thermal scenario described above: 43 ± 5 MPa and 12 ± 2 MPa for CF/PEEK with PEI of 250 µm and 50 µm, respectively.

Summary

This study focused on the temperature profiles during welding for different thicknesses of ED. CF/PEEK parts with 250 µm and 50 µm thick PEI layers were assembled by USW. A non-intrusive experimental procedure was carried out to measure the temperature at the welding interface. It was observed that the thermocouples must be inserted entirely inside the layer, otherwise, they can alter the temperature measurement.

The curves of power and displacement of the sonotrode over time have shown that different stages and specific points can be identified (heating, flowing of PEI, final flowing and cooling) in comparison with the temperature profiles. As the interface is a closed contact during welding, measuring the temperature at the interface is difficult. However, welding power and displacement have been shown to have a strong connection with thermal history. Too high power during welding distorts the composite.

To evaluate the thermal diffusion from the interfaces between CF/PEEK and PEI layers, a FEM was developed. Considering the input parameters for the model, it is important to mention that thermal contact resistance has a significant impact on thermal diffusion. For both thicknesses, the PEI reaches its T_g, allowing macromolecular interdiffusion between the two parts, the condition to get a strong weld. Besides, the model predicts that only the first ply of the composite parts is thermally affected, in which the temperature within the PEEK matrix exceeds its T_m. Such local heating at the interface prevents composite degradation and deconsolidation. However, in the case of the smallest ED thickness, a too-high power in a short time leads to poor welding of APC-2 parts. It has been confirmed with the mechanical characterization of welds, 12 MPa was obtained for the 50 µm PEI layer, compared to 43 MPa obtained for 250 µm. A higher PEI thickness at the interface slows down the cooling step of highly conductive unidirectional CF/PEEK composites, resulting in a deeper interdiffusion of PEI and so, a stronger weld. These results contribute to a better understanding of the thermal transfer phenomena during USW. By varying the ED thickness, we demonstrated the possibility to assemble composite parts by USW without affecting the whole composite parts, thus preventing composite degradation and deconsolidation.

Further work will focus on PEEK and other polyaryletherketones (PAEK) as energy directors, to assess the effect of crystallinity at the interface on mechanical strength.

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