Pre-Heating of Thermoplastic Sandwich Materials for Rapid Thermoforming

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ABSTRACT: The thermoforming of thermoplastic sandwich structures based on the same matrix polymer for the face sheets and the core requires precise temperature control both before and during forming. Two opposing requirements must be satisfied: the face sheets must be heated to above the glass transition temperature of the polymer, T_g, whereas the temperature of the core should not approach T_g in order to avoid the collapse of the foam cells. A numerical model was used to determine the optimal thermal parameters for thermoforming using an inverse method. The conductivities of the skins, the interface, and the foam were first calculated by the inverse method and then implemented in a thermal analysis. It was shown that the thermal requirements for the skins and the foam cannot all be fulfilled using a classic one-step heating route; therefore, a two-step procedure was proposed. The numerical tool allowed the principal heating parameters, namely the temperature of the heating plates and the holding and transfer times, to be determined.

KEY WORDS: thermoforming, PEI, thermoplastic sandwich, process window, thermal model, inverse method, thermo-physical properties, processing parameters.

INTRODUCTION

ALTHOUGH THERMOFORMING IS an unconventional process for manufacturing sandwich parts [1], it offers the potential for short cycle times and large series production. Recent studies have been published on the subject of thermoformable

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sandwiches made with a 3D-knitted core [2], a honeycomb core [3,4], or a core and face sheets based on different thermoplastic matrices [5,6]. However, the literature on the thermoforming of sandwiches based on the same thermoplastic matrix for both the face sheets and the core is limited.

Two major phenomena must be taken into consideration for the thermoforming of thermoplastic sandwiches. First, the occurrence of folds and wrinkles in the face sheets during forming is a direct consequence of the reinforcement architecture. To this end, investigations were carried out on different fiber textiles. Tensile tests performed in various directions on woven and knitted fabrics showed that whereas woven fabrics are highly anisotropic and accommodate the deformation by the trellis effect, knitted fabrics exhibit larger deformations and a far more isotropic behavior due to the local stretching of the loops. Drapability tests were performed to relate the forming energy to the preform architecture. Due to its high drapability and low forming energy, a double warp-knitted bar knit was selected and impregnated with a polyetherimide (PEI) thermoplastic matrix [7,8]. Mechanical tests at forming temperatures were carried out on these knitted laminates. The results showed that the knitted laminates are highly deformable with strains of up to 70% and isotropic properties, which makes the forming of defect-free parts possible [9,10]. Consequently, the problem of wrinkling can be overcome using these knitted laminates, and they have been used as face sheets for thermoformable sandwiches.

Second, the preheating of these sandwiches before forming needs special attention. Various studies have been published on the prediction of temperature in composite laminates during preheating [11–14], but limited information was found on the heating of sandwich structures. These sandwich configurations provide inherently very good thermal insulation, which can complicate heating control [15]. Furthermore, for a sandwich made of the same thermoplastic matrix for the face sheets and the foam core, two opposing requirements must be satisfied: the face sheets must be heated to above the glass transition temperature, T_g, of the matrix, whereas the foam core should be heated only to temperatures below T_g to avoid the collapse of the foam cells [16]. Studies of the thermal behavior of the PEI knitted reinforced face sheets at high temperatures have shown that the minimum forming temperature at which forming is practical is around 280°C [10]. Tensile and compressive tests performed on PEI foam showed that the forming temperature window ranges from 165°C to 185°C [9]. Figure 1 shows the two processing windows for the foam and the face sheets and the required temperature profile through the sandwich section. Therefore, it was necessary to develop a heating procedure adapted to these sandwiches, together with precise control of the forming temperature. These aspects are treated in the present study.

To predict the process feasibility and to determine the optimal preheating conditions while avoiding a trial-and-error experimental approach, for which the measurement of the in-situ temperature in the thin skins and at the core-skin interface

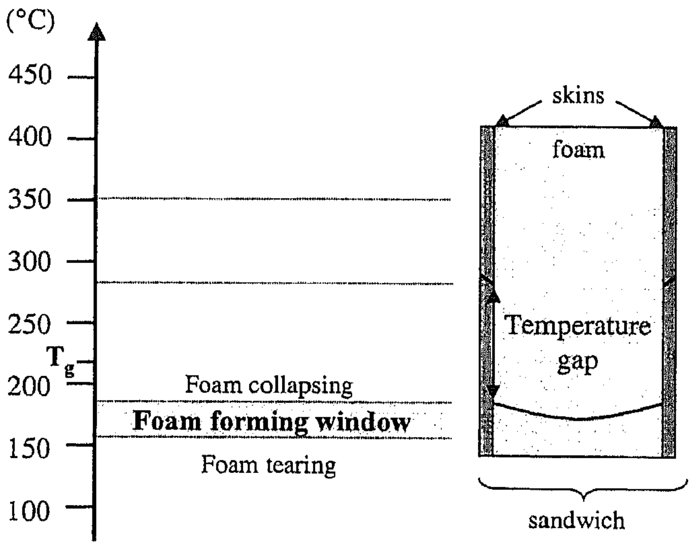

Figure 1. Schematic thermal process window of PEI knitted laminates (sandwich skins) and PEI foam (sandwich core). For optimal thermoforming, a thermal gradient must be created through the sandwich thickness.

would be difficult, a mathematical model was developed to calculate the temperature profile in flat thermoplastic sandwiches. The thermo-physical properties of the skins and foam core, as well as the boundary conditions resulting from the contact heating and from the cooling in air during transportation, were required to model these temperature profiles. Both were determined using an inverse method, and they were then implemented into a thermal heat flow code (CalcoMOS) [17], as presented and discussed below.

DETERMINATION OF PROPERTIES AND BOUNDARY CONDITIONS BY INVERSE METHOD

Principle

The idea of the inverse method is based upon minimization of the errors between calculated and measured temperatures at given positions and times. This approach is the numerical equivalent solution to a standard least-squares analytical method [18]. For a series of m thermocouples inserted inside a sandwich at various precisely defined locations, x_j ($j = 1, m$) and used to measure at times, t_i ($i = 1, n$) the temperatures, T_{ij}^m, then the error function to deduce a set of parameters $P = \{P_1, P_2, ..., P_q\}$ can be written as [19]

$$\varepsilon(P) = \sqrt{\sum_{i=1}^{n}\sum_{j=1}^{m}(T_{ij}^m - T_{ij}^c(P))^2} \tag{1}$$

where $T_{ij}^c(P)$ is the calculated temperature at a location j at a time i. By iterations, the error is minimized by adjusting the q components of the vector P. When the maximum relative variation of the parameters P is smaller than a desired tolerance, the calculation is finished and the solution accepted. Determination of the thermo-physical properties and boundary conditions is briefly presented below.

Experimental

MATERIALS

Polyetherimide (PEI) was used for the foam core and for the face sheets. This amorphous polymer belongs to the polyimide family, and its T_g is 217°C. It has a very high temperature resistance, is auto-extinguishable, and produces no toxic fumes [20,21]. PEI reinforced laminates used for the sandwich skins were reinforced with one layer of glass-fiber double warp-knitted bar fabric. The thickness was 0.75 mm, and the fiber volume fraction V_f was 27.5%. The 10-mm thick PEI foam panels used for the core had a density of 80 kg/m^3 and are manufactured by Airex AG under the trade name R 82.80. The bonding of the face sheets to the core was realized with an epoxy film of 150 g/m^2 commercialized by Hexcel under the trade name Structufilm R-382 H.

DIFFERENTIAL SCANNING CALORIMETRY

Differential scanning calorimetry (DSC) experiments were carried out to determine the PEI matrix and PEI foam heat capacities. Experiments were conducted on 10-mg samples heated from 25°C to 350°C at a heating rate of 10°C·min^{-1}. The heat capacity was determined by a derivation from the measured enthalpy, ΔH.

MEASUREMENT OF TEMPERATURE PROFILES THROUGH THE THICKNESS

To apply the inverse method and check the model validity, measurements of the temperature at well-defined locations through the sandwich thickness were carried out. The skins and foam core thickness were modified and enlarged up to 10 mm for the skins and 40 mm for the foam, to enable the measurement of temperature. Thus, these modified thermoplastic sandwich specimens, 140 mm long and 60 mm wide, were equipped with sixteen thermocouples distributed through the thickness. Five thermocouples were inserted within the face sheet thickness, one on each side of the epoxy bonding film, and seven thermocouples were inserted through the foam thickness. Each thermocouple was horizontally shifted by 10 mm to reduce the influence of the neighboring thermocouple. The one-dimensional heat flow through the structure was checked by adding two more thermocouples at the same vertical distance from the interface.

DETERMINATION OF THE BOUNDARY CONDITIONS

The thermal boundary conditions are dictated by the heating by contact with the hot plates and then the cooling during transportation and were determined with the help of an inverse method. Temperature histories during heating and cooling in air were recorded in the skin and the foam of modified sandwiches. The boundary condition at the end of the one-dimensional model was a Dirichlet condition at one extremity, where the temperature history recorded at that location was imposed, and a Cauchy condition at the other extremity, where the heat flux was assumed to be

$$q = -h \cdot (T_s - T_{ext}) \tag{2}$$

where h is the heat transfer coefficient, T_s the surface temperature of the sandwich, and T_{ext} the temperature of the air surrounding the sandwich. An initial guess for h was assumed, and iterative calculations were made so that the difference between the computed and measured temperature histories at the locations in between the two extremities of the domain become as small as possible. Note that q is negative when cooling in air ($T_s \geq T_{ext}$) and positive during the heating step ($T_s \leq T_{ext}$).

Results

Figure 2 compares the computed and measured temperature evolutions at the thermocouple locations during heating. It can be observed that the calculated points are in very good agreement with experimental results. When convergence was attained, the quadratic error found between the measured and predicted data was always less than 1°C. The specific heats and thermal conductivities, respectively measured and deduced, are listed in Table 1.

Figure 3 compares the computed and measured temperature evolutions during

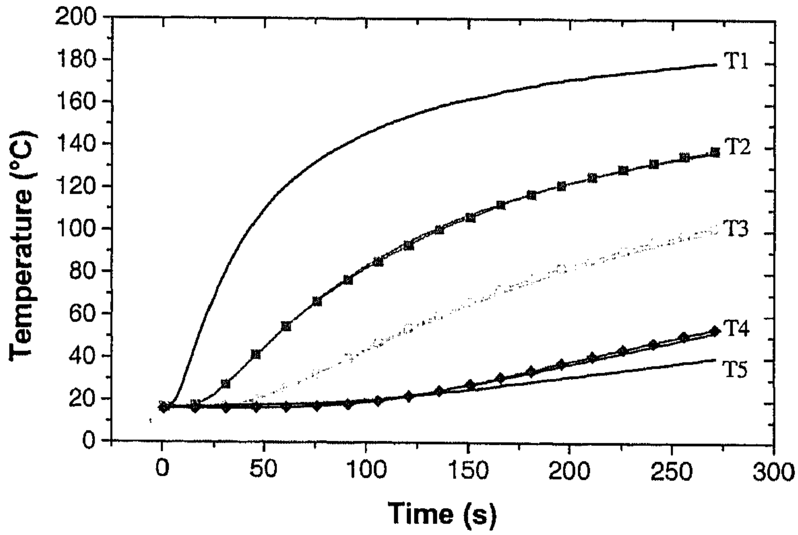

Figure 2. Comparison between computed (solid lines) and measured (lines with symbols) temperature evolutions at the locations situated between the two extreme thermocouples (results obtained with the foam).

Table 1. Material thermo-physical properties used as inputs for the simulation.

Component	Density (g/cm³)	Thermal Conductivity *k* (W/m·K)	Specific Heat, C_P (J/kg K)
PEI knitted skins	1.62	0.1725	911.1 + 2.7 * *T*
PEI foam core	0.80	0.0364	734.7 + 4.6 * *T*

The specific heats were measured with differential scanning calorimetry while the thermal conductivities were deduced using an inverse method.

cooling. The heat transfer coefficient determined for the air cooling phase is given in Table 2 with the quadratic error at convergence.

Determination of the heat transfer coefficient by contact heating with hot plates was less straightforward, since it depends on the face sheet thickness. Therefore, it was not possible to use the inverse method to deduce the missing coefficient of the skins, since an accurate value of the temperature gradient through laminates less than 0.7 mm in thickness is difficult to determine. On the other hand, it was possible to record a single temperature measurement for the skins and to fit the missing parameter to calculate the real temperature profile. Comparison of several heating experiments made on knitted sandwiches enabled an average value of the heat transfer coefficient to be deduced (Table 2). The high value of *h* is explained by a

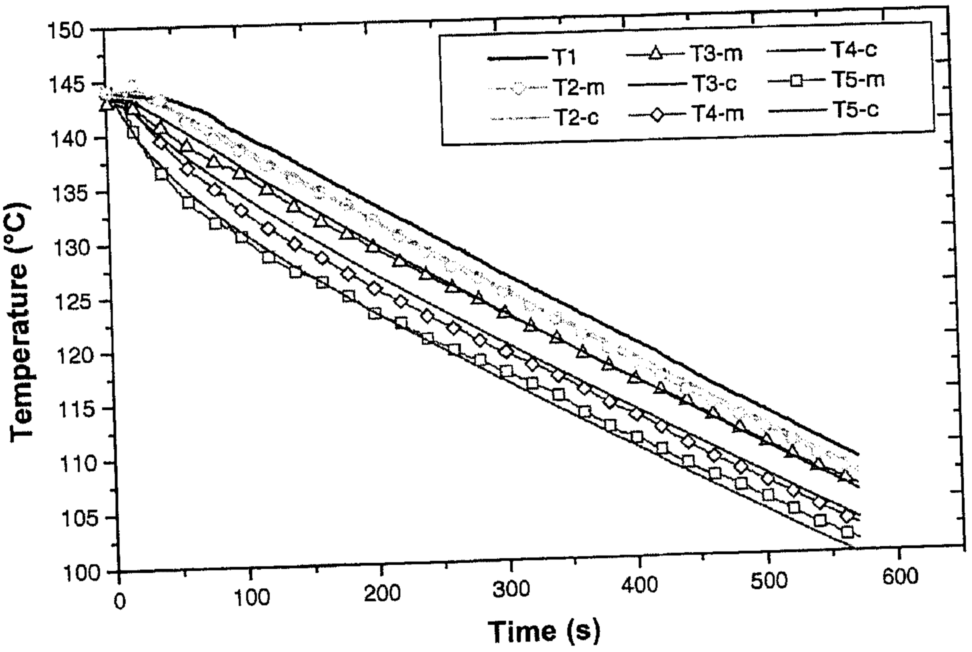

Figure 3. Comparison between computed (Ti-c) and measured (Ti-m) temperature evolutions at the thermocouple locations during cooling of a knitted reinforced face sheet.

Table 2. Heat transfer coefficients and quadratic errors at convergence for hot plate contact heating and cooling in the air.

	Air Cooling	Heating with a Hot Plate
h (W/m^2 K)	16	10,000
T_{ext} (°C)	20	320
Error (°C)	0.672	—

good contact between the skins of the sandwich [22] and the heating plates and by the pressure exerted on these hot plates [23,24].

THE HEATING PROCEDURE

The conductive heat transfer technique was used because it offers high rates of heating and a homogeneous heat distribution over the sandwich surfaces. Two different heating schemes were investigated, namely one-step and two-step techniques.

Heating in One-Step

Pre-heating experiments were done using only one heating step. In that case, the sandwich structure was heated from room temperature by contact with hot plates as shown in Figure 4. The thermal model developed was used to calculate the temperature profiles throughout the sandwich using the one-step heating route. The evolution of the temperature profiles during heating with hot plates at 280°C is given in Figure 5. The predicted results confirmed the experimental observations and the fact that while the face sheets were heated within their forming window temperature, the core temperature was too low in the middle and too elevated in the vicinity of the interface. Consequently, the one-step heating method was not suitable.

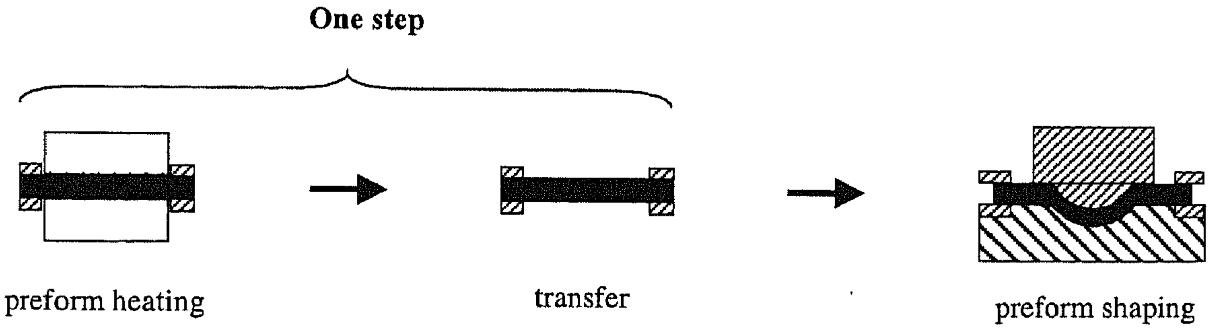

Figure 4. Schematic of a classic heating procedure in one step.

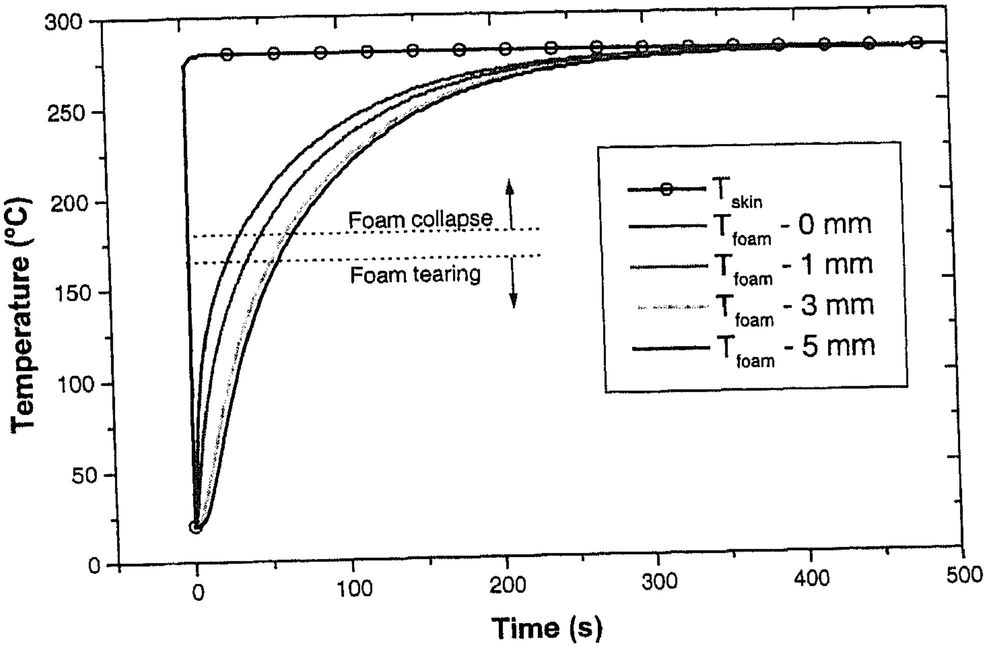

Figure 5. Temperature evolution through the section of a knitted sandwich using hot plates at 280°C during the one-step heating procedure. The temperature profiles required within the foam prior to forming cannot be achieved.

The Two-Step Heating Technique

A transient thermal field in the sandwich structure leads to a temperature difference between the skin and the core owing to the finite value of the heat conductance between the two media. This condition was used to fulfill the thermal requirements by using a new heating procedure herein called the *two-step heating technique* and illustrated in Figure 6. The entire sandwich structure is first heated between two hot plates at the lower forming temperature of the foam. Once thermal equilibrium is achieved, the sandwich is transferred to a second set of plates held at the forming temperature of the skins. The aim of this second heating step is to heat only the skins to their forming temperature, while ensuring that the foam is at its minimum forming temperature through its entire thickness. Hence, control of the foam temperature in this second step is independent of the foam panel thick-

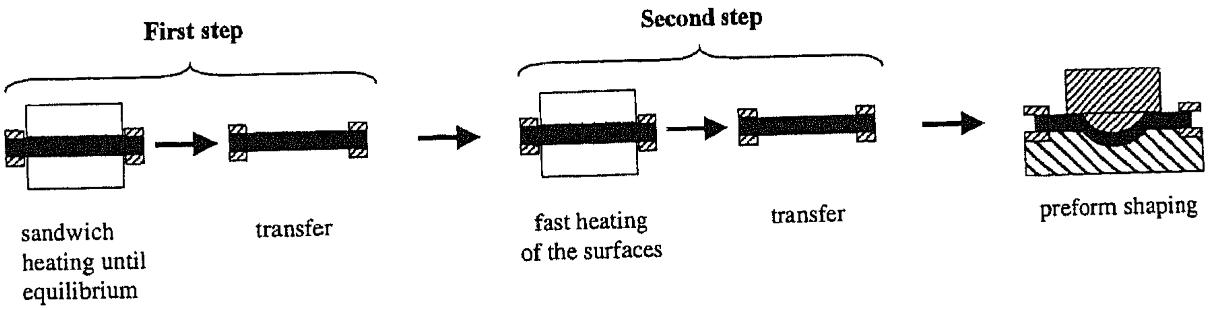

Figure 6. Schematic of the retained heating technique in two steps.

ness, which is often varied according to structural and bending stiffness requirements.

THERMAL MODEL OF THE TWO-STEP HEATING TECHNIQUE

The FEM Model

An FEM model of the two-step heating technique was developed with the commercial code CalcoMOS. The material properties and the boundary conditions described above were implemented in the thermal model. The real geometry of the sandwich structure was used. Due to symmetry reasons, only half of the sandwich thickness was modeled under the assumption of a one-dimensional heat transfer. The lower frontier of the domain was adiabatic (zero heat flux), whereas on the top, time-dependent boundary conditions are imposed. The thermal model was first used to determine the holding time required to heat the sandwich to 165°C during the first stage of the two-step heating procedure. As represented in Figure 7, the thermal equilibrium through the sandwich section is reached after 380 s. The main benefit offered by this model was the ability to study the influence of the main process parameters on the heating of the sandwich prior to forming.

Effect of the Main Parameters

To determine the optimum heating parameters, a systematic approach was used. The transportation time between the two heating devices (including the clos-

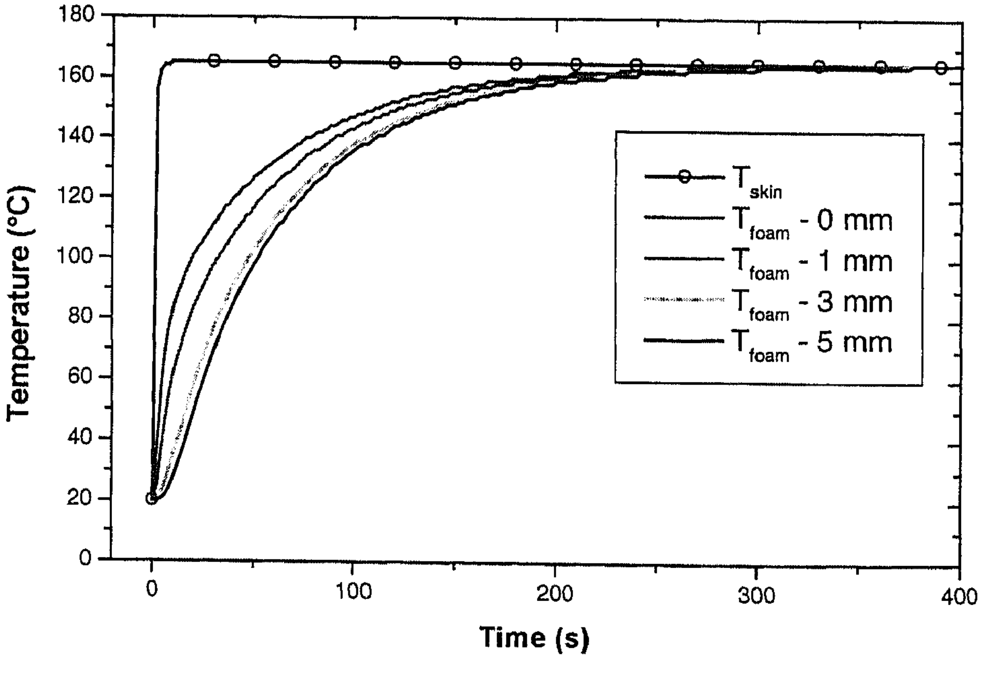

Figure 7. Temperature evolution through the knitted skinned sandwich during the first heating stage. The uniform temperature of 165°C is reached after 380 s.

Table 3. Systematic variation of the hot plate temperature and holding time of the second heating stage to study the influence of the variation of each parameter on the temperature profile of the knitted sandwiches.

	Holding Time (s)		
Temperature (°C)	**2.5**	**5**	**10**
280		*K* - 1	
300	*K* - 2	*K* - 3	*K* - 4
320		*K* - 5	

ing of the hot plates) was set to 2 seconds. This is a limiting time for the transfer. Similarly, the transfer time to the press, which includes the mold closure, was kept constant and equal to 2 seconds. The holding time and the plate temperature in the second heating stage were varied according to the values presented in Table 3. By varying the important parameters along the main column and row of values, it was possible to determine their influence and thus to deduce rapidly the optimal parameters.

Results and Discussion

The influence of the heating temperature on the temperature profiles of knitted sandwiches at a constant holding time of five seconds is plotted in Figure 8. The final temperature of the skins was significantly influenced by the hot plate temperature. As the heating temperature was increased, the final temperature of the skins

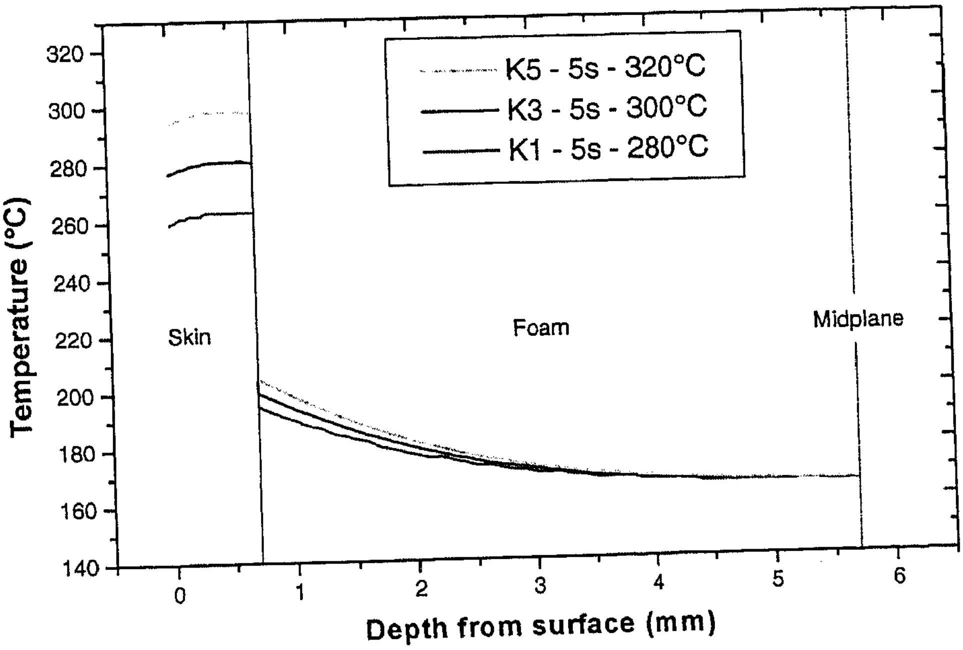

Figure 8. *Influence of the hot plate temperature on the knitted sandwich temperature profile at a constant heating time of 5 seconds.*

increased. It is interesting to observe that the final temperature of the foam was affected to a much lesser degree than that of the skins, even in the vicinity of the interface. This phenomenon is explained by the low thickness of the skins in comparison to the foam, the higher conductivity of the skins, and the heat resistance of the interface, which dictates the thermal gradient inside the sandwich. To achieve the two thermal requirements in the skins and in the foam concurrently, it is suggested that a high plate temperature should be used to keep the skins above their minimum forming temperature.

The influence of the holding time on the temperature profiles through knitted sandwiches at a process temperature of 300°C is shown in Figure 9. The final temperature through the foam thickness was fairly affected by the variation of the holding time. When the holding time was increased, the foam temperature became higher, especially in the interface area. The final temperature of the skins is not significantly controlled by a change in the holding time. As above, the thermal behavior of the sandwich is explained by the difference of at least one order of magnitude between the thickness and the conductivity of the skins and the core. To favor the sandwich forming with respect to its processing window temperature, it is advisable to use a short holding time to maintain the foam below its maximum forming temperature.

To conclude the present analysis, it is suggested that a temperature above 320°C be used to keep the skins above their minimum forming temperature, and a short holding time less than 5 s to keep the foam below its maximum forming temperature. The temperature evolutions at various locations within the skins and the foam for the knitted sandwiches using 320°C and 5 s for the hot plate temperature

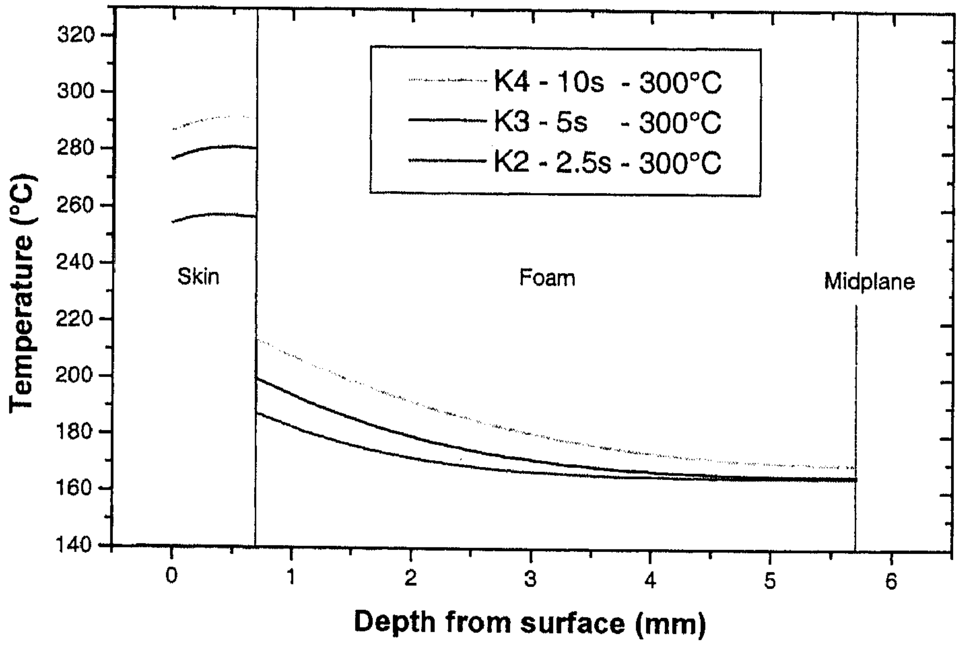

Figure 9. Influence of the heating time on the knitted sandwich temperature profile at a constant hot plate temperature of 300°C.

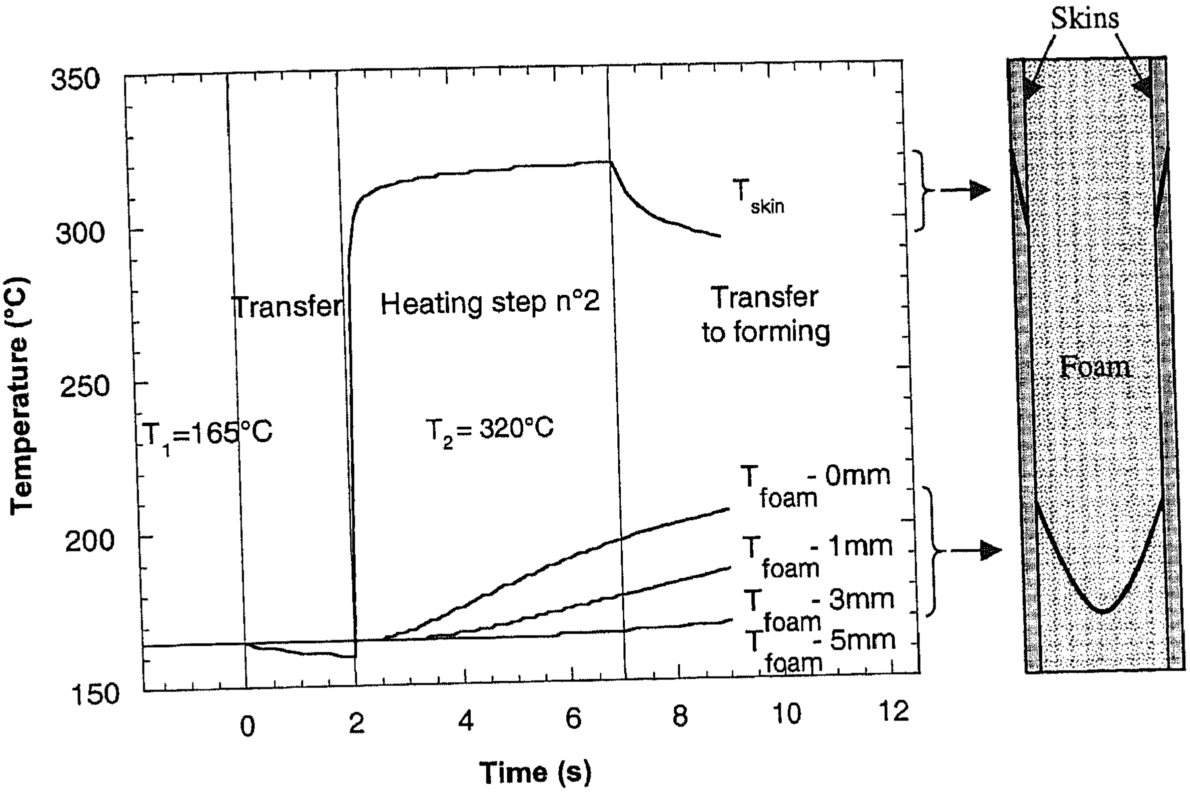

Figure 10. Calculated temperature profile evolution for a PEI knitted sandwich with a 10-mm-thick core. The thermal gradient required for thermoforming is achieved using the optimal processing parameters.

and holding time are given in Figure 10. During the transfers, the skins cooled down immediately, while the foam temperature still increased for several seconds. This result is explained by the fact that the interface heat conductance is a barrier to the energy transfer towards the foam core. Thus, the skins and the foam are in their respective forming window temperatures. In conclusion, the thermoforming of PEI knitted sandwiches is conceptually possible using the temperature and process times explained above. Thus, a heating device that allows fast and precise heating cycles was developed, and thermoformed sandwich parts with double curvature were manufactured.

CONCLUSIONS

The thermoforming of PEI sandwich preforms was studied. The heterogeneous nature of the sandwich structure, in particular the presence of an interface between two different materials and the associated heat resistance, was used to fulfill the specific thermal requirements prior to the forming stage. The face sheets must be in a softened state above T_g, while the foam should not exceed the temperature below T_g at which collapse is initiated. An FEM model was developed to simulate the temperature profiles in the sandwich during the heating stage. Thermal properties of the sandwich components, determined by an inverse method, as well as appropriate boundary conditions were implemented into the heat flow model. It was shown that the thermal requirements could not be fulfilled by heating the structure in one step. Therefore, a two-step heating cycle was used. This solution guarantees easy control of the minimum foam forming temperature and generates a strong temperature gradient between the face sheets and the foam core. The main parameters governing the heating cycles are the holding time under the second heating stage, the temperature of the hot plate, and the transfer times. For simplification, the transfer times between the first and second heating stages and between the heating set-up and the press were kept constant. Systematic variations of the main parameters showed that the holding time preferentially influences the temperature of the foam, while the plate temperature essentially controls the temperature of the skins. The optimal holding time and hot plate temperature were determined so that the sandwich structure would have the required temperature profile just prior to thermoforming. Thermoformed sandwich structures were manufactured successfully using those processing conditions.

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