Microwave Assisted Resin Transfer Moulding

M.S. Johnson BS, MS, PhD, C.D. Rudd[1] BSc, PhD, CEng, FIMechE, FIM, D.J. Hill[2] BEng, PhD
Department of Mechanical Engineering, The University of Nottingham

Abstract
Traditionally, RTM has been limited to low volume production due to protracted component cycle times. The principal cause of extended cycle times is thermal quench near the injection gate, occurring when cold resin enters the heated mould. The period required for the mould and resin to recover the lost heat and cure, lengthens the cycle time. One method to decrease thermal quench and reduce the cycle time is to preheat the resin prior to injection. A specialised in-line microwave resin preheating system has been developed for this purpose. A small volume of resin, having a low thermal inertia, passes through the in-line system, affording a rapid heating response to variations in the input microwave power. Resin can be heated accurately to a constant, pre-defined temperature using this system. Furthermore, prescribing an analytical heating function allows profiling of the resin temperature during the injection phase. The cure sequence can be controlled using a ramping profile, with the additional benefit of a lower resin viscosity for improved flow through the mould. As a result, impregnation and cycle times are reduced considerably.

1. Introduction
High volume production of reinforced polymeric composites (more than 100,000 parts per annum) has been limited by excessive manufacturing and material costs, recycling difficulties, and the lack of a reliable manufacturing technique. Resin transfer moulding (RTM) could overcome some of the difficulties associated with the production of thermoset-based FRP components at medium to high volume levels [1]. RTM involves injecting a liquid thermosetting resin into a heated, closed mould containing a dry fibre preform. Air and excess resin are purged through peripheral vents. The part is removed from the mould after completion of the curing process and finishing operations are performed as necessary. Injecting cold resin into the hot tool quenches the mould and laminate temperature. This effect is most pronounced in the gate region where thermal recovery of the mould and laminate is suspended until the end of impregnation. Subsequently, the mould heating system raises the temperature of the mould body and this heat is transferred to the resin by conduction, initiating the exothermic cure reaction. Completion of the cure sequence is signalled by cure at the injection gate. This is a direct consequence of the thermal quench in that region and the variation in chemical "age" across the laminate. Process developments involving zone heated moulds [2] and phased initiator resin systems [3] have been used to compensate for thermal quench and have provided significant reductions in the cycle time. Preheating the resin prior to injection is an alternative technique in this respect and provides additional operating advantages including viscosity reduction. This paper describes the application of a purpose built, in-line microwave system to preheat resin and reduce the RTM cycle time.

2. Characteristics of the RTM Process
Thermal and pressure histories provide a means for identifying key events in the non-isothermal RTM cycle [4]. The present study is based upon the experimental RTM facility at the University of Nottingham, shown schematically in Figure 1. The facility contained an electroformed nickel shell mould for the production of FRP automotive undershields to protect the engine and gear box from heavy impact damage under rally conditions. Four thermocouples were positioned in the upper mould half at positions 2, 3, 4, and 5, protruding into the mid-plane of the mould cavity. Similarly, four pressure

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[1] To whom correspondence is to be directed

[2] Now at the Ford Motor Company, Dearborn, MI 48121 USA

transducers were located opposite the thermocouples in the lower mould. Resin entered the mould though a centrally located pin gate at position 5.

Kendall et al. [5] characterised the in-plane thermal history during the manufacture of a liquid moulded component. Additional experimentation by Lebrun et al. [6] has contributed information on the through thickness laminate temperature variations in a flat moulded component. These studies provide an understanding of the factors governing the non-isothermal cycle in addition to the validation of numerical methods currently being used to model the non-isothermal injection moulding process [7]. A typical thermal history is shown in Figure 2 for a "hot" preform injected with ambient temperature resin. The cycle time begins when resin arrives at the injection gate and ends after completion of the exothermic cure reaction. The impregnation phase is defined as the period from resin arrival at the injection gate (distinguished by a sudden drop in temperature at position 5) to a rise in the gate temperature after the injection valve is closed. The period available for fibre preform wet out and interfacial bond formation is approximated by the time to the first peak exotherm, occurring near the mould periphery (position 2). Cool resin contacting the hot mould surface produces a sharp drop in temperature (thermal quench) at that location. Light-weight shell moulds have a low thermal mass and are particularly susceptible to quench. The injection gate remains quenched until the end of impregnation at which time heat is recovered gradually and the resin activation temperature is reached. The resin cures last at the injection gate, dictating the cycle time, as a result of thermal quench and a comparatively short residence time. A steady-state temperature distribution is developed near the injection gate during thermal quench. Tucker [7] has developed an analytical solution for this similar, idealised case as a benchmark for the numerical solution.

Figures 3a and 3b show a typical pressure history for resin injected at ambient temperature into a heated mould. The pressure rises at the injection gate during impregnation and stabilises after impregnation is completed. Resin shrinkage indicates the onset of cure, producing a drop in pressure. Thermal expansion during the exothermic reaction leads to a subsequent pressure increase. This discrete sequence is evident at the mould periphery (position 2). Closer to the mould centre (position 3), the pressure increases after the hydrostatic phase but before resin shrinkage and the subsequent exothermic pressure rise. The increase in pressure before shrinkage occurs to a greater degree near the injection gate. This rise in pressure, termed the pre-exotherm pressure [5], is attributed to the thermal expansion of the liquid resin. Resin at the mould periphery cures first creating a rigid mould seal, entrapping a pool of uncured resin and air within the mould. Thermal expansion of the resin during the exothermic cure reaction transmits a pressure through the liquid pool. Compression of any residual air within the liquid pool increases as the cure front progresses inward, generating a high localised pressures near the injection gate. The intensity of the pre-exotherm pressure near the gate overshadows the exothermic pressure rise making it imperceptible at positions 4 and 5. It is because of this effect that the local pre-exotherm pressures on the mould are critical design loads, rather than the hydrostatic pressure which is typically lower for RTM [8].

3. Microwave Resin Preheating

Preheating the resin before it enters the mould is one means of reducing thermal quench. The heat required to initiate cure is decreased, resulting from a lower temperature difference between the resin and the mould. The low thermal conductivity of polymer resins (typically 0.3 W/mK [9]) makes heating significant quantities by conventional means difficult. Conduction heating techniques are likely to produce a large thermal gradient which, for reactive resin systems, would lead to premature cure at the heat transfer surface. Microwave heating of low conductivity materials is attractive since this dielectric heating process is essentially volumetric, providing uniform heating throughout the product. Exposing a dielectric material, such as a polymer resin, to an alternating electric field at microwave frequencies causes its dipoles to become polarised. The energy required to rotate the dipoles to a polarised state is stored within the dielectric material. When the electric field is reversed, the dipoles relax to an equilibrium position before being re-polarised by the alternated electric field. The release of stored energy during the dipolar relaxation produces heat [10]. The temperature rise in the dielectric material is expressed as [11]:

$$\Delta T = \frac{2\Pi f E^2 \varepsilon_o \varepsilon''}{\rho C_p} t \qquad (1)$$

It can be seen from Equation 1 that the temperature rise in the material is a function of the electric field which depends, in turn, on the input power.

Microwave heating for domestic and industrial applications normally uses a multimode resonant applicator. Microwaves are reflected off the walls of the applicator, combining with incident microwaves to form a standing wave, termed a mode. A multimode applicator supports several coincident modes that interact to produce a non-uniform modal structure. A mode includes both electric and magnetic field components, but only the electric field component contributes to dielectric heating. For this reason, future reference to a mode will concern only the resonating electric field, being equivalent to the heating profile within the applicator. Maximum heating occurs when the product is situated at a mode peak (antinode), with zero heating at a node. The uneven modal distribution within a multimode applicator produces a non-uniform heating profile. As a result, localised hot and cold spots develop within multimode applicators in relation to the mode amplitude at particular locations [12].

A single mode can be excited by changing the size and shape of the applicator to specific dimensions. Knowledge of the mode shape permits location of the resin at the antinode for maximum heating. Since the balance of microwave power is directed into a single mode, heating is more concentrated than for multimode ovens. Single mode applicators typically are rectangular or cylindrical and are classified further according to the electromagnetic field patterns that they produce. Field patterns for unique applicators are defined as having either a transverse electric (TE) field or a transverse magnetic (TM) field orientation. The TE and TM modes are subclassified by three subscripts l, m, and, n which denote the number of half wavelengths occurring along each coordinate direction. A comprehensive treatment of electromagnetic field pattern nomenclature is given by Mariner [13].

Microwave heating of FRP materials has been applied primarily to curing of thermosets. Strand [14] used a multimode microwave oven for this purpose, achieving a thirty fold reduction in cure time over conductive heating. Boey [15] modified an autoclave for microwave curing of glass reinforced epoxy components. Laminates were placed in a single-sided mould within the autoclave. A PC controlled, microwave emitting horn, was passed over the surface of the component with dosage levels being controlled by variations in the microwave power and the traversing speed of the horn. Methven and Ghaffariyan [16] used an in-line TM_{010} mode cylindrical applicator to cure FRP rod during pultrusion. Similarly, Cassagnau and Michel [17] used an in-line TE_{01n} mode rectangular applicator to polymerise ethylene vinyl acetate (EVA) and ethylene methyl acrylate (EMA) in a microwave transparent polypropylene matrix. The authors claimed that the morphology of the structure could be controlled by varying the residence time within the applicator. Microwave batch preheating has been investigated by Costigan and Birley [18], who demonstrated that by preheating a charge of sheet moulding compound (SMC), resistance to flow was reduced and a lower capacity press could be used. Furthermore, the temperature drop across the mould was reduced, lowering the cycle time. Batch preheating of polyester resins using a multimode microwave oven for RTM was reported by Rudd [19] who demonstrated a cycle time reduction of 20% in the manufacture of small plaques.

An in-line TM_{020} mode cylindrical applicator has been used in this study and the electric field distribution for this device is shown in Figure 4. The variable heating profile across the diameter of the applicator allows a single stream of reactive resin to be preheated. Low injection pressures (typically less than 10 bar) associated with RTM result in flow rates of the order of 5 l/min giving rise to a laminar resin velocity profile within the injection line. An even heating profile across the PTFE pipe would overheat the resin at the pipe wall. The heating profile of the TM_{020} mode cylindrical applicator is analogous to the laminar flow profile, producing zero heat at the PTFE pipe wall where the resin is stagnant, and maximum heat along the pipe axis where the velocity is greatest. Risman and Ohlsson designed such a microwave system to heat thermally sensitive viscous fluid slurries in a TM_{021} mode resonant cylindrical applicator [20]. Based upon this work, Hill developed the in-line TM_{020} mode resonant cylindrical applicator to preheat polyester resin [21]. The applicator and the PTFE pipe diameters were dictated by the dielectric properties of the elements within the applicator (resin, PTFE, and air).

The continuous nature of the in-line microwave resin preheating system complies with high volume production strategies. Rapid heating is possible due to the low thermal inertia associated with the small resin volume, and minimal wastage is produced. Precise control of the preheating system allows time-wise variations in the resin temperature to be implemented during the injection phase. Conversely, batch preheating does not provide precise control of the resin temperature. Furthermore, bulk storage of preheated, initiated resin is impractical as its reactivity increases at elevated temperatures. Flushing of the injection system between consecutive mouldings would be necessary to avoid premature cure.

4. The Experimental RTM Facility and Microwave Preheating Equipment

A comprehensive description of the design, operation, and control of the RTM facility (Figure 1) has been given by Kendall [22]. Prototype engine undershield components were made using a light-weight, electroformed nickel shell mould. The 6 mm mould halves were bonded to cast aluminium backings then mounted onto stiffeners to constrain mould shell deflections. The upper tool was lowered evenly using synchronised hydraulic jacks at each corner of the stiffener while the lower tool remained stationary. Ten peripheral clamps held the mould closed. Mould heating was by hot oil which circulated through copper piping embedded in the backing material of each mould half. A 48 kW heater and a 15 kW chiller unit regulated the oil temperature. The constant pressure injection system consisted of a 30 litre resin storage vessel which could be pressurised up to 7 bar using a laboratory air supply. A single stream of initiated resin passed through 4 m of flexible hose before reaching the centrally located pin gate in the lower mould half. The resin was contained within the mould cavity by a closed cell, silicone foam seal around the mould circumference. Air and excess resin were purged through two vents at the periphery of the lower mould half.

Figure 1 also shows the in-line microwave system installed within the moulding facility to preheat a single stream of reactive resin during injection. A cylindrical applicator operating in the TM_{020} mode was attached to a microwave generator (magnetron) with WG9A waveguide. The water cooled magnetron operated at a nominal frequency of 2.45 GHz and was adjustable over a 0.5-5.0 kW power range. Microwave energy generated in the magnetron was directed through the rectangular waveguide (internal section - 86 mm x 43 mm) to the cylindrical applicator. A circulator was installed between the magnetron and the applicator to prevent reflected microwave energy from damaging the magnetron during use. Reflected microwaves were transferred to a stream of cooling water (water load) that passed continuously through the circulator. The microwave was operated automatically in sequence with the moulding process. A digital to analog convertor (DAC) board was installed within the personal computer (PC) that was linked to a programmable logic controller (PLC). A digital switching facility on the card turned the microwave ON and OFF during the moulding sequence. Microwave power was adjusted through an analog channel on the board via a proportional-integral-derivative (PID) controller. A feedback control loop based on the resin outlet temperature and incorporating the PID controller compensated for a reduction in the resin flow rate during impregnation due to increased back pressure in the mould. A unique resin set point temperature could be specified prior to injection. In addition, a temperature profile, based upon the mass of resin injected, could be prescribed.

The moulding cycle was controlled automatically by the same PC and PLC system. A load cell measured a specific amount of reactive resin to be drawn into the pressure vessel. The moulding sequence was initiated remotely using the PC. The mould was lowered and held closed by the peripheral clamps. The storage vessel was pressurised and injection began. A capacitive proximity sensor located just beyond the microwave outlet indicated the presence of resin within the microwave applicator and signalled it to be turned ON. Similarly, vent proximity sensors indicated resin arrival at those locations. The microwave was switched OFF and injection ceased after resin reached both vents on either side of the mould. The pneumatically operated injection and vent valves were flushed immediately with solvent to prevent heated resin at those locations from curing. Thermal and pressure histories were displayed on the PC monitor throughout the moulding cycle. The mould opened after the resin had cured, completing the automatic cycle.

5. Constant Temperature Resin Injection

Two series of mouldings were produced with resin injected at a constant elevated temperature to determine the effect on the RTM cycle. The mould was heated to 60°C and 90°C for each series. A different initiator was used at each mould temperature preventing a direct comparison between the two studies, but a qualitative analysis could be performed based upon the established trends.

5.1 Constant Resin Temperature Injection into a 60°C Mould

Components were produced using the in-line microwave resin preheating system at a mould temperature of 60°C. A benchmark moulding was made with polyester resin [Synolac 6345 initiated with 2% bis t-butyl peroxy dicarbonate (Perkadox 16)] injected at ambient temperature (22°C). Subsequent mouldings were produced with resin preheated to 30°C, 40°C, 45°C, and 50°C. Successive increases in the resin temperature were expected to reduce the impregnation and cycle times. Since the resin preheat temperature was always less than the mould temperature, quench occurred in every case.

Impregnation and Cycle Time Effects

Figure 5a shows the relationship between impregnation time and resin preheat temperature. Raising the resin temperature from 22°C to 40°C reduced the impregnation time by 41%. Increasing the resin temperature to 45°C caused no additional reduction in the impregnation time, while a further temperature increase to 50°C extended the impregnation time by 14 seconds. This resulted from a rise in viscosity within the mould prior to the end of injection, as indicated by the flow of coagulated resin from the mould vents. Figure 5b shows that increasing the resin temperature from 22°C to 40°C reduced the cycle time by 24%. An additional 10°C increase did not reduce the cycle time any further as demonstrated by a levelling of the curve.

The above results suggest that for the resin system and moulding parameters used in this series of trials, 40°C was the optimum resin preheat temperature. This arises from the interaction of resin viscosity and gel time, both of which are temperature dependent. Figure 6 shows the relationship between viscosity and temperature for Synolac 6345 resin. A 70% reduction in resin viscosity occurs when heating the resin from 20°C to 40°C. Heating the resin further from 40°C to 50°C reduces the viscosity by only an additional 40%. The 70% reduction in viscosity between 20°C and 40°C improved resin flow through the mould and preform, decreasing the impregnation time (Figure 5a). The proportional relationship between the impregnation time and resin viscosity corresponded with the fill time expression for radial flow [5]:

$$t_{fill} = \frac{\Phi\mu}{2KP_{gate}} R_m^2 \left(\ln \frac{R_m}{R_{gate}} - \frac{1}{2} \right) \quad (2)$$

The reduction in viscosity was diminished over the 40°C to 50°C temperature range. Figure 7 shows that by increasing the resin (II) temperature from 40°C to 50°C significantly reduces the initiation time. The consequence of a reduced initiation time became apparent at a resin preheat temperature of 50°C as localised high viscosity regions developed within the mould prior to the end of injection. Resin flow was impeded as a result, and the impregnation time (Figure 5a) increased by 12%.

Figure 8 shows the values of log fill time (Figure 5a) and log initiation time (Figure 7, resin II) plotted against the resin preheat temperature. The initiation time curve should cross the fill time curve at 40°C, signifying that the optimum resin preheat temperature had been reached. This would indicate that the resin was near its initiation temperature, and preheating to a higher temperature would promote gel within the mould prior to complete filling. Such information could be used to construct a mouldability diagram as described by Gonzalez-Romero and Macosko [23], defining a process window for the impregnation phase. Resin initiation for this investigation (Figure 7) was defined as the time required for the resin temperature to rise 5°C above the oil bath temperature in which it was immersed. It is expected that significant resin conversion had occurred prior to reaching the +5°C temperature. As a result, the actual initiation times reported in Figure 8 were inappropriate for predictive use, and a more sensitive technique to measure the increase in resin viscosity as a function of temperature would be required.

Thermal Effects

Figure 9 shows the peak exotherm temperatures along the mould diagonal (positions 2-5) at the five different resin preheat temperatures. Conventional RTM was represented by injecting resin at ambient temperature (22°C) with peak exotherm temperatures ranging from 106°C at the mould periphery (position 2) to 119°C at the injection gate (position 5). Injecting resin at 50°C did not increase the peak exotherm temperatures significantly, suggesting that resin preheating did not alter the kinetics of the cure reaction. Figure 10 presents further evidence of this showing thermal histories at the injection gate with resin injected at 22°C and 50°C. Increasing the resin temperature to 50°C reduced the quench by 23°C and resulted in a 24% reduction in cycle time. However, resin injected at 22°C and 50°C both cured at approximately the same rate as indicated by the similarity in shape of the two thermal histories above the mould temperature (60°C). This suggests that conventional RTM moulds designed to operate below a thermal damage threshold, such as GRP or epoxy backed moulds, can be used safely with resin preheating to reduce cycle times.

Pressure Effects

Figure 11 shows the peak pressures measured within the mould along the diagonal (positions 2-5) at five different resin preheat temperatures. Two additional mouldings were manufactured at 23°C and 50°C with the pressure being recorded at the injection gate. An extreme pressure variation was measured across the mould diagonal ranging from 1 bar at the mould periphery (position 2) to 26 bar at the injection gate (position 5). These high pre-exotherm pressures were identified by Kendall [22] as a characteristic of centre gate injection. Like conventional RTM, constant temperature resin injection had no effect on the cure sequence so that high pre-exotherm pressures were measured at all elevated resin temperatures. However, the peak pressures did not differ significantly from those produced during conventional RTM, suggesting that microwave resin preheating could be used without causing mould damage.

5.2 Constant Resin Temperature Injection into a 90°C Mould

A second series of mouldings was produced using the in-line microwave resin preheating system at a mould temperature of 90°C. A benchmark moulding was made using polyester resin [Synolac 6345 initiated with 1% t-butyl peroxy 2-ethyl hexanoate (TBPEH)] injected at ambient temperature (20°C) followed by four others at 30°C, 40°C, 50°C, and 55°C. A qualitative comparison of the trends established at both moulding temperatures (60°C and 90°C) was expected to provide a general understanding of the effects of constant resin temperature injection.

Impregnation and Cycle Time Effects

Figures 12a and 12b show the effect of resin preheat temperature on the impregnation and cycle times. Increasing the resin temperature from 20°C to 55°C reduced the impregnation time by 50% and the cycle time by 29%. Unlike the results obtained at a 60°C mould temperature (Figure 5b), the cycle time continued to decrease as the resin preheat temperature was increased. Figure 12b indicates that the impregnation time also decreased as the resin preheat temperature was increased. The rate of reduction in the impregnation time, however, was less between the higher resin injection temperatures. Referring to Figure 7, this result suggests that at 55°C the resin system (III) was far removed from its gel temperature so that slight viscosity reductions continued to reduce the impregnation and cycle times. It was predicted that these values would plateau when the resin temperature reached an initiation time near 20 minutes (75°C) as occurred at the 60°C mould temperature (Figures 5a and 5b). However, raising the resin temperature required additional microwave power, which in turn increased the likelihood of premature cure within the cylindrical applicator. For this reason, the optimum resin preheat temperature could not be demonstrated for this series of mouldings.

Thermal and Pressure Effects

Figures 13a and 13b show the peak exotherm temperatures and the peak cavity pressures along the mould diagonal (positions 2-5) at the five different resin injection temperatures. Conventional RTM was represented by injecting resin at ambient temperature (20°C). The trends are similar to those in Figures 9 and 11 produced at a 60°C mould temperature, although, a direct comparison is difficult since equivalent resin systems were not used. Peak exotherm temperatures (Figure 13a) did not increase with increasing resin injection temperature, suggesting that the relationship between the peak exotherm temperature and the resin preheat temperature was independent of the resin formulation. Development of high pressures near the injection gate (Figures 11 and 13b) was demonstrated at both mould temperatures (60°C and 90°C). This result was anticipated since high pre-exotherm pressures were associated with the converging cure front.

5.3 Discussion of Constant Resin Temperature Injection

Several benefits of in-line resin preheating at constant temperature have been demonstrated. Impregnation and cycle times were reduced as a result of a lower resin viscosity and thermal quench. Average cycle time reductions of 27% were realised without increases in the peak temperatures or pressures within the mould during the cure phase. These results seemed to occur independently of the resin formulation. In addition, resin preheating was not expected to alter the cure kinetics of the resin system. The results imply that a constant temperature resin injection system could be retrofitted in a conventional RTM facility to reduced cycle times without damaging the mould. Isothermal impregnation to eliminate thermal quench would reduce cycle times further.

6. Isothermal Impregnation in RTM

A moulding was produced under isothermal impregnation conditions to demonstrate the relationship between resin residence time within the mould and cycle time. Figure 14 shows the thermal histories at the mould periphery (position 2) and the injection gate (position 5). The mould and preform temperature stabilised at 42°C prior to injection. The microwave was turned on after resin reached the injection gate proximity sensor, downstream of the microwave applicator, thus a small volume of resin that initially entered the mould was not preheated. As a result, the laminate temperature dropped to 34°C (position 5) before stabilising at 40°C after 12 seconds of heating. Cure occurred first at the mould periphery after 402 seconds, and last at the injection gate for a cycle time of 580 seconds.

Neglecting the initial quench, the first resin into the mould travelled to the periphery at a constant temperature of 40°C. The residence time for the last resin to enter the mould (40°C) at the injection gate commenced with the end of injection. The difference in resin residence time at the mould periphery compared to resin at the injection gate equalled the impregnation time (164 seconds). The time difference between the peak exotherms at the gate and periphery (178 seconds) should have equalled the impregnation time. Initial quench may have delayed the onset of cure at the mould periphery, accounting for the slight variation.

Excessive cycle times have been attributed to quench near the injection gate, as both the mould and resin must be reheated to the nominal mould temperature before the onset of cure. As a result, resin always cures last at the injection gate, since the cure front advances from the periphery towards the centre of the mould. However, this same cure sequence was generated under isothermal conditions in the absence of thermal quench, with the cycle time still being dictated by cure at the injection gate. This result occurred due to a difference in the resin residence time within the mould at elevated temperatures. Resin positioned at the mould periphery had a the longest residence time and cured first, whereas resin cured last at the injection gate as its residence time within the mould was shortest. Ramping the resin temperature during injection was investigated to minimise the cycle time by equalising the chemical "age" of the resin and thereby altering the cure sequence.

7. Ramped Resin Temperature Injection

A benchmark moulding, representing conventional RTM, was made by injecting polyester resin [Synolac 6345 initiated with 2% acetyl acetone peroxide (Trigonox 44B) and 0.5% cobalt accelerator (NL49P)] at ambient temperature (24°C) into a mould heated to 40°C. Four additional mouldings were produced by ramping the resin temperature as follows: 40-45°C, 40-50°C, 40-55°C, and 40-60°C. The preheating sequence was based upon a linear ramping of the resin set point temperature as a function of the total mass of resin injected. The set point temperature was initialised to 40°C at the start of injection (0 kg injected), and increased to the maximum temperature by the end of injection (9 kg injected).

Thermal and Pressure Effects

The effect of resin temperature ramping on the moulding thermal histories is shown in Figures 15a and 15b. Insignificant changes in the time to peak temperature resulted at the mould periphery (435 seconds on average) as shown in Figure 15a. Resin at that position was preheated to 40°C, and experienced isothermal conditions. These results agreed closely with the isothermal moulding shown in Figure 14 which had a peak exotherm time of 402 seconds at the mould periphery. The time to peak exotherm at the injection gate (position 5) decreased with successive increases in the resin temperature gradient. Like the constant resin temperature mouldings, ramping the resin temperature did not increase the value of the peak exotherm temperature significantly as can be seen in Figure 15b.

Figure 16 illustrates that the magnitude of the pre-exotherm pressures decreased as the ramping rate increased. Subsequent pre-exotherm pressure values were measured at the injection gate (22°C and 40-50°C) to illustrate the trend at that location.

Discussion of Ramped Resin Temperature Injection

Ramped resin temperature injection allowed the cure sequence across the mould to be controlled by varying the ramping rate. Conventional RTM was represented by injecting resin at ambient temperature (24°C), demonstrating the typical cure sequence, from the periphery to the centre of the mould. Separation between the first and last peak exotherm temperatures for the conventional moulding was

268 seconds as shown in Figure 17a. Ramping the resin temperature from 40-45°C during injection reduced the separation to 68 seconds. Applying a temperature ramp from 40-50°C led to coincident resin cure across the mould surface with a 36% reduction in cycle time over the moulding produced by conventional RTM as shown in Figure 17b. Virtually no separation (13 seconds) existed between the exotherm peaks at the injection gate and mould periphery. Increasing the temperature ramp further reduced the time to peak exotherm at the injection gate, and led to a reversed cure sequence from the centre to the periphery of the mould as shown in Figure 17c. However, since the time to peak exotherm at the mould periphery remained unchanged (435 seconds on average), no further reduction in cycle time was realised (Figure 15a). The condition of coincident cure resulted in the lowest cycle time possible for the specific resin system and moulding parameters.

Pre-exotherm pressures within the mould cavity increased along the diagonal from the mould periphery to the injection gate for mouldings having a typical cure sequence (Figure 16, 24°C resin injection temperature). Ramping the resin temperature to promote coincident cure reduced this pre-exotherm pressure to the hydrostatic level as no entrapment of the liquid pool occurred. This suggests that mould deflections were reduced. Consequently, tool life can be extended while thickness variations in the laminate due to mould deflection can be minimised using the in-line microwave resin preheating system.

8. Conclusions

Non-isothermal moulding conditions resulted in thermal quench at the injection gate, dictating the component cycle time. An in-line microwave resin preheating system was installed to compensate for quench within the mould. This system allowed continuous processing and was compatible with high volume production methods. Rapid heating and cooling of resin was possible with the in-line microwave system due to the low thermal inertia associated with the small resin volume. Preheating resin to a constant temperature below the mould temperature decreased the quench and resin viscosity, leading to reduced cycle times. Isothermal moulding conditions eliminated the quench entirely, but cure at the injection gate continued to govern the cycle time due to a minimum resin residence time at that location. A typical cure sequence commencing at the mould periphery and advancing toward the injection gate typified these mouldings. A characteristic of this cure sequence was high pre-exotherm pressures near the injection gate. Ramped resin temperature injection was demonstrated, producing coincident resin cure across the mould. Resin at the injection gate was preheated above the mould temperature to initiate cure more rapidly, and compensate for the minimal residence time. Coincident resin cure reduced the cycle time by 36%, and represented the minimum cycle time possible for the given resin system and moulding parameters. In addition, coincident resin cure eliminated the high pre-exotherm pressures at the injection gate.

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Nomenclature

C_p	specific heat of the dielectric material (J/kg °C)
E	magnitude of the electric field (V/m)
f	frequency (nominally 2.45 GHz)
K	reinforcement permeability (m^2)
P_{gate}	pressure at injection gate (Pa)
R_{gate}	injection gate radius (m)
R_m	mould radius (m)
t	period of microwave heating (s)
ΔT	temperature rise of the dielectric material (°C)

ε_o	dielectric permittivity of free space (8.8542 x 10^{-12} F/m)
ε''	relative loss factor of the dielectric material
μ	resin viscosity(Pa s)
ρ	density of the dielectric material (kg/m^3)
Φ	porosity

References

[1] **C. D. Rudd, M. J. Owen, K. N. Kendall, and I. D. Revill.** "Developments in Resin Transfer Moulding for High Volume Manufacture," pages 301-314. *American Society of Materials/Engineering Society of Detroit, Advanced Composites Conference, Detroit, MI USA, October 1990.*

[2] **M. J. Owen, V. Middleton, K. F. Hutcheon, F. N. Scott, and C. D. Rudd.** "The Development of Resin Transfer Moulding (RTM) for Volume Manufacture." *Proceedings of the Institution of Mechanical Engineers, Design in Composite Materials* pages 107-114, 1989.

[3] **M. S. Johnson, P. J. Blanchard, and C. D. Rudd.** "Cycle Time Reductions in RTM Based on Resin Preheating and Phased Initiators," pages 291-296. *Proceedings of the 7th European Conference on Composite Materials, London, UK, May 1996.*

[4] **C. D. Rudd and K. N. Kendall.** "Towards a High Volume Manufacturing Technology for Composite Components." *Proceedings of the Institution of Mechanical Engineers Journal of Engineering Manufacture* **206** pages 77-91, 1991.

[5] **K. N. Kendall, C. D. Rudd, M. J. Owen, and V. Middleton.** "Characterisation of the Resin Transfer Moulding Process." *Composites Manufacturing* **3**(4) pages 235-249, 1992.

[6] **G. Lebrun, R. Gauvin, and K. N. Kendall.** "Experimental Investigation of Resin Temperature and Pressure during Filling and Curing in a Flat Steel RTM Mould." *Composites Part A* **27A** pages 347-355, 1996.

[7] **C. L. Tucker III.** "Heat Transfer and Reaction Issues in Liquid Composite Molding." *Polymer Composites* **17**(1) pages 60-72, 1996.

[8] **C. D. Rudd and K. N. Kendall.** "Thermo-Mechanical Effects During Resin Transfer Moulding." *Plastics, Rubber and Composites Processing and Applications* **22**(4) pages 223-233, 1994.

[9] **G. Kaye and T. Laby.** "Tables of Physical and Chemical Constants and Some Mathematical Functions." 15th edition, Longman Group Ltd., pages 287-290, 1986.

[10] **W. R. Tinga and S. O. Nelson.** "Dielectric Properties of Materials for Microwave Processing-Tabulated." *Journal of Microwave Power* **8**(1) pages 23-27, 1973.

[11] **R. C. Metaxas and R. J. Meredith.** "Industrial Microwave Heating." Peter Peregrinus Ltd., 1983.

[12] **D. C. Dibben and R. C. Metaxas.** "Finite Element Time Domain Analysis of Multimode Applicators Using Edge Elements." *Journal of Microwave Power* **29**(4) pages 242-251, 1995.

[13] **P. F. Mariner.** "Introduction to Microwave Practice." Heywood & Company, pages 18-20, 1961.

[14] **N.S. Strand.** "Fast Microwave Curing of Thermoset Parts." *Modern Plastics* pages 64-67, October 1980.

[15] **F. Y. C. Boey, I. Gosling, and S. W. Lye.** "Processing Problems and Solutions for an Industrial Automated Microwave Curing System for a Thermoset Composite," pages 651-656. *The 9th International Conference on Composite Materials, Madrid, Spain, July 1993.*

[16] **J. M. Methven and S. R. Ghaffariyan.** "The Design and Performance of a Single Mode Resonant Cavity as a Heating Device in Pultrusion," Paper 2 pages 1-5. The Plastics and Rubber Institute. *Proceedings of the 3rd International Conference on Automated Composites, The Hague, Netherlands, October 1991.*

[17] **P. Cassagnau and A. Michel.** "Continuous Crosslinking of Ethylene Vinyl Acetate and Ethylene Methyl Acrylate Copolymer Blends by On-Line Microwave Heating." *Polymer Engineering and Science* **34**(12) pages 1011-1015, 1994.

[18] **P. J. Costigan and A. W. Birley.** "Microwave Preheating of Sheet Moulding Compound." *Plastics and Rubber Processing and Applications* **9**(4) pages 233-240, 1988.

[19] **C. D. Rudd, M. J. Owen, and V. Middleton.** "Effects of Process Varibles on Cycle Time During Resin Transfer Moulding for High Volume Manufacture." *Materials Science and Technology* **6** pages 656-665, July 1990.

[20] **P. O. G. Risman.** "Method of Preparing Foodstuffs Containing Coagulated Proteins and a Device for Performing the Method." *US Patent 4,237,145*, 1980.

[21] **D. J. Hill.** "Microwave Preheating of Thermosetting Resin for Resin Transfer Moulding." PhD thesis, University of Nottingham, 1993.

[22] **K. N. Kendall.** "Mould Design for High Volume Resin Transfer Moulding." PhD thesis, University of Nottingham, 1991.

[23] **V. M. Gonzalez-Romero and C. W. Macosko.** "Design Strategies for Composite Reaction Injection Molding and Resin Transfer Moulding," pages 1292-1295. The Society of Plastics Engineers. *45th Annual Technical Conference-ANTEC'86, Boston, MA USA, 1986.*

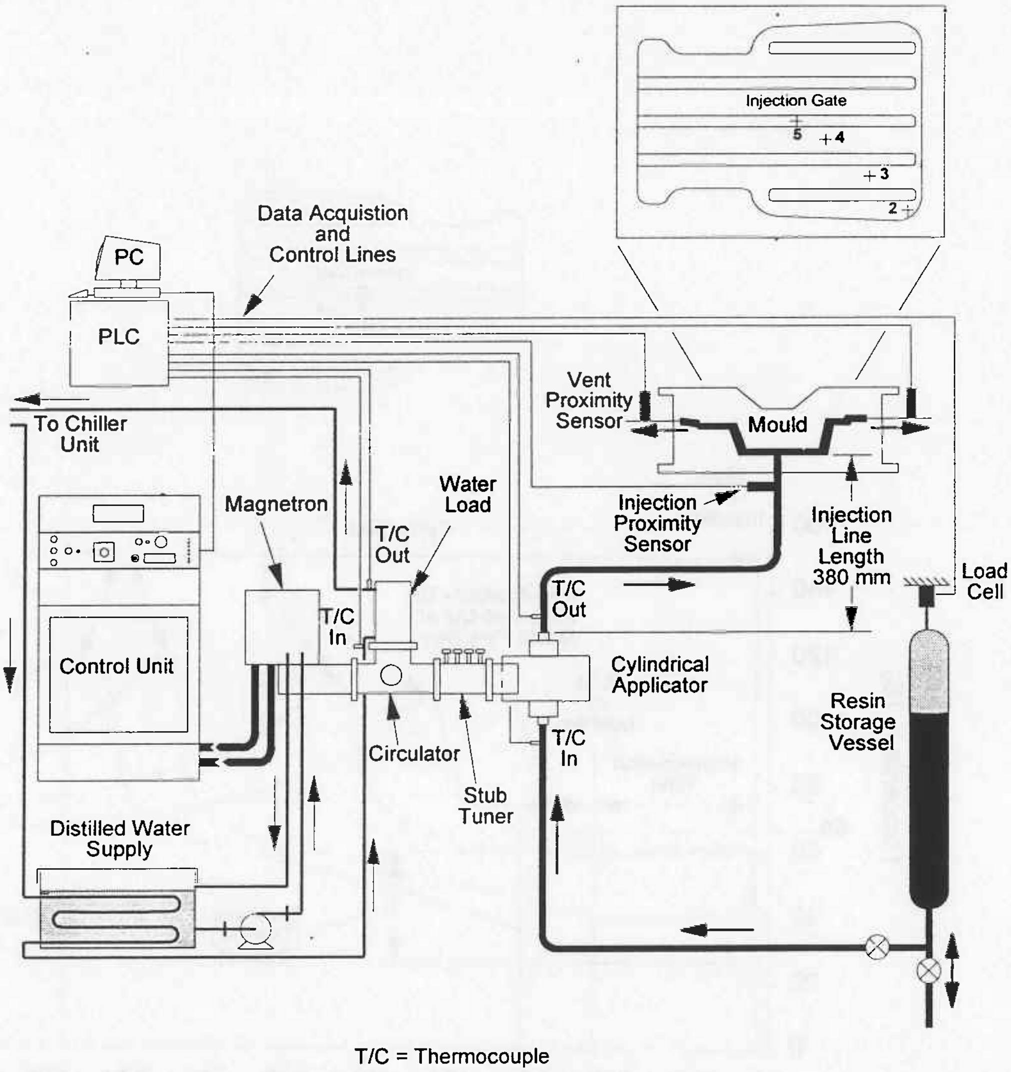


Figure 1 Schematic of Facility for microwave assisted RTM

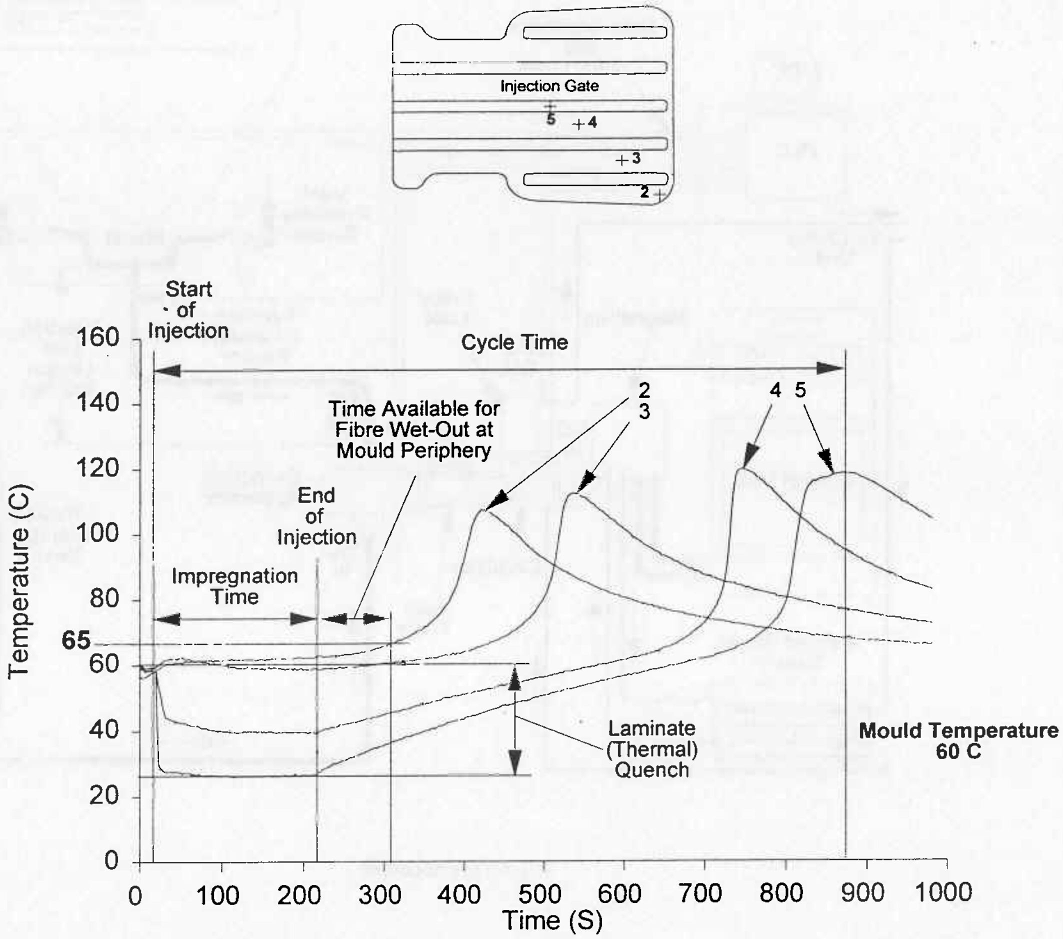


Figure 2 Thermal history of the moulding cycle showing principal events

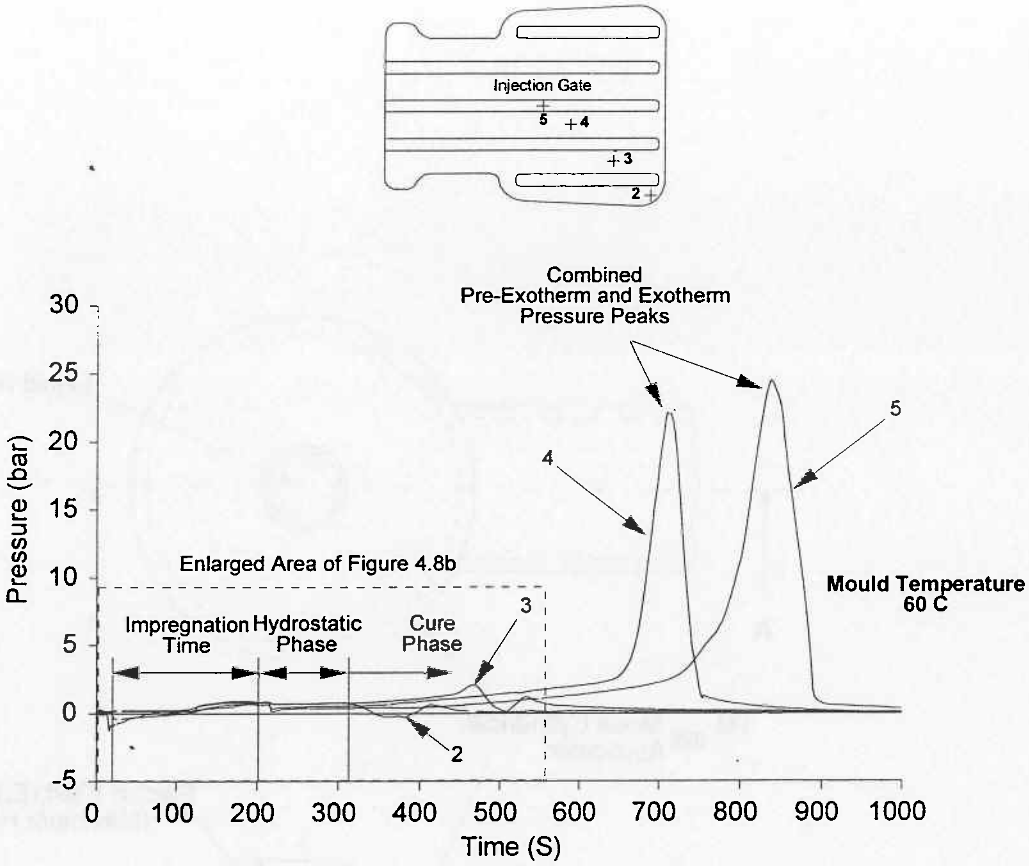


Figure 3a Pressure history of the full moulding cycle.

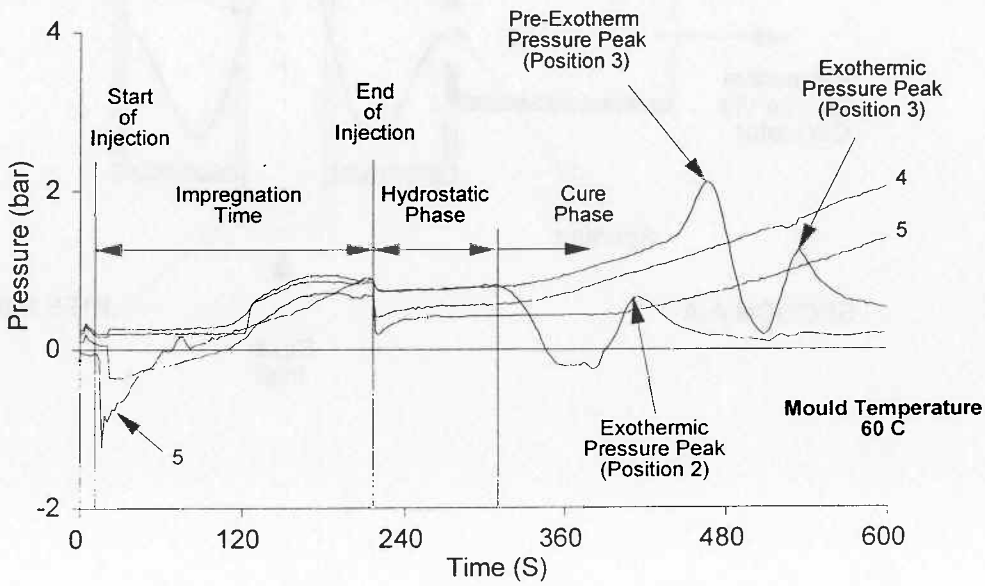


Figure 3b Pressure history during the first 600 seconds of the moulding cycle showing principal events

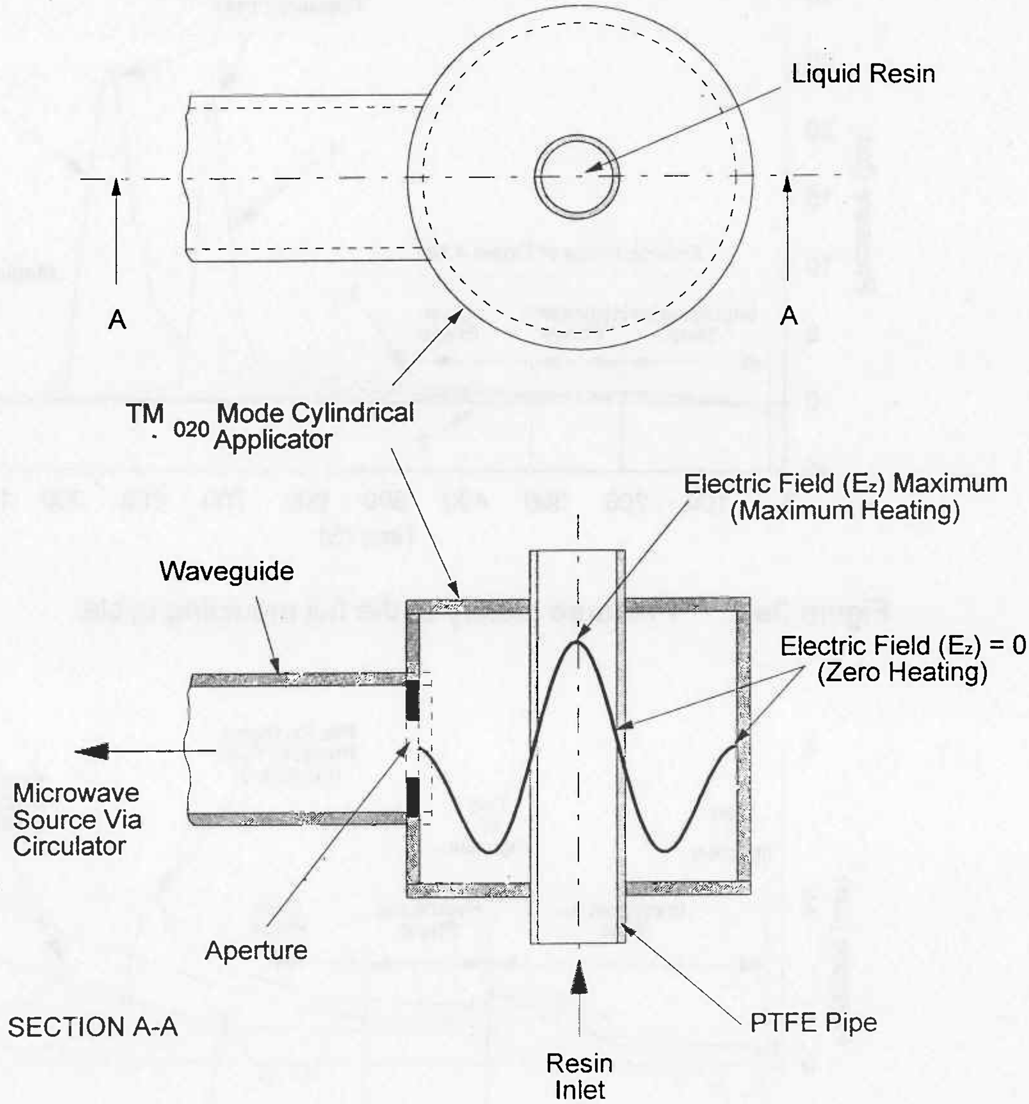


Figure 4 TM_{020} mode cylindrical applicator

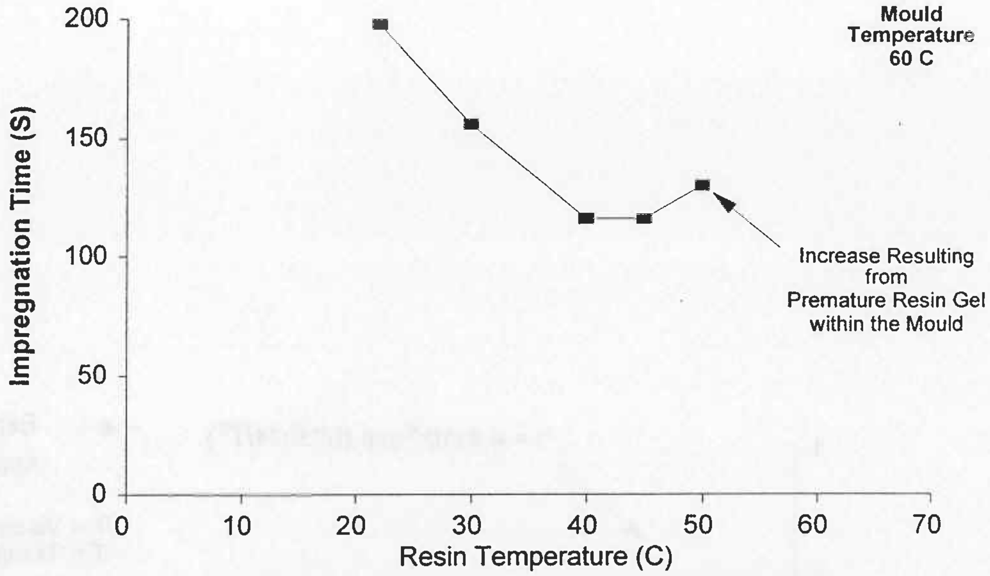


Figure 5a Effect of resin temperature on impregnation time for undershield components produced at a mould temperature of 60 C

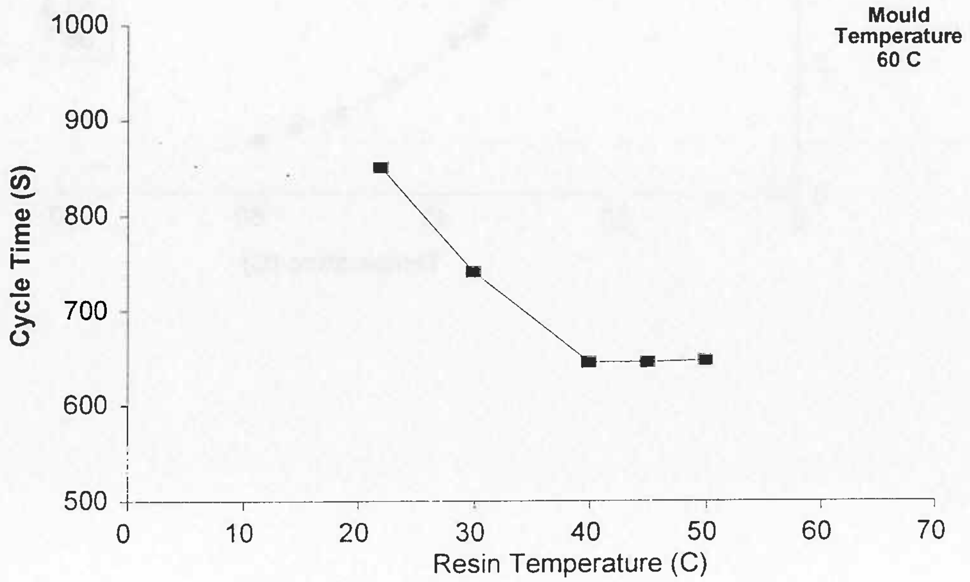
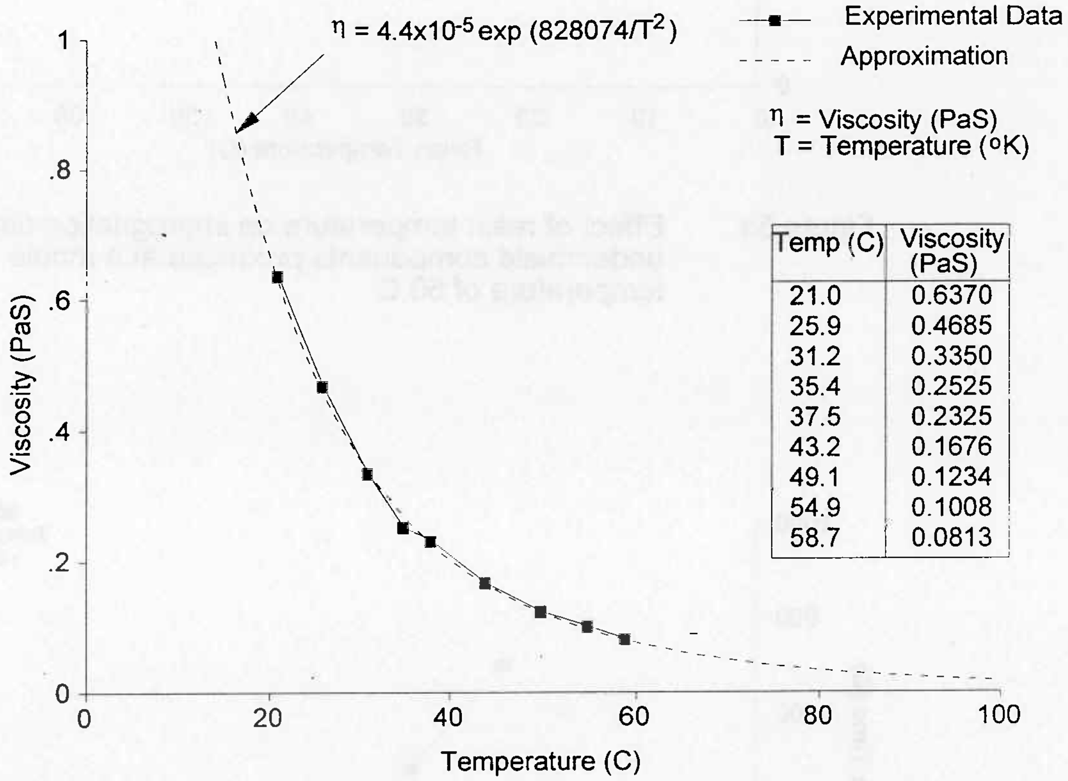
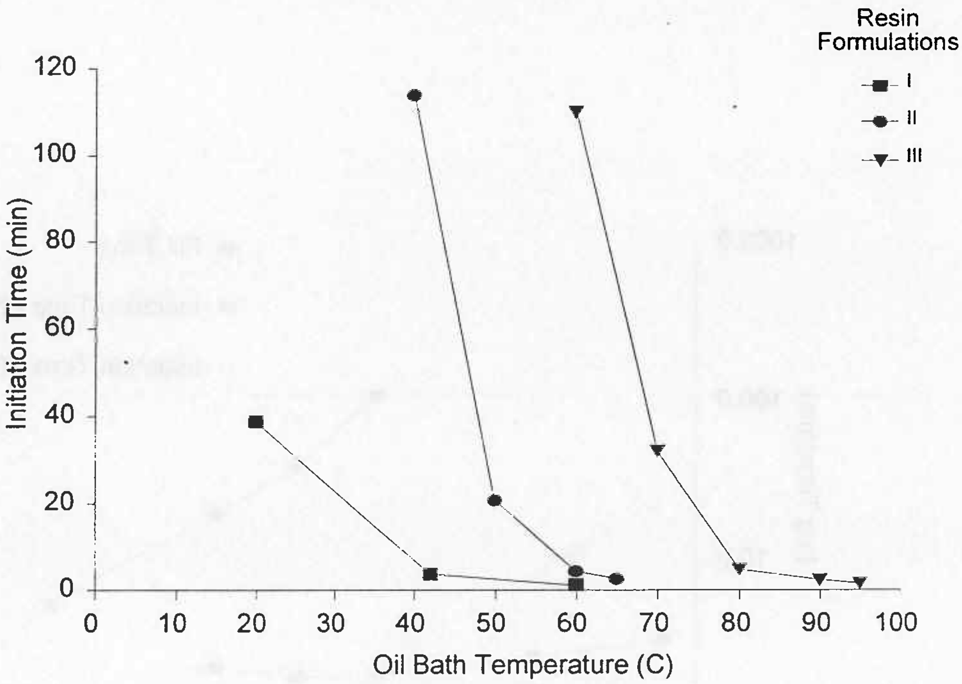


Figure 5b Effect of resin temperature on cycle time for undershield components produced at a mould temperature of 60 C



Temp (C)	Viscosity (PaS)
21.0	0.6370
25.9	0.4685
31.2	0.3350
35.4	0.2525
37.5	0.2325
43.2	0.1676
49.1	0.1234
54.9	0.1008
58.7	0.0813

Figure 6 Viscosity versus temperature of Synolac 6345 polyester resin



Key to Resin Formulations:

I	CVP 6345 + 2% Trigonox 44B + 0.5% NL49P
II	CVP 6345 + 2% Perkadox 16
III	CVP 6345 + 1% TBPEH

Figure 7 Initiation time versus temperature for the experimental resin systems

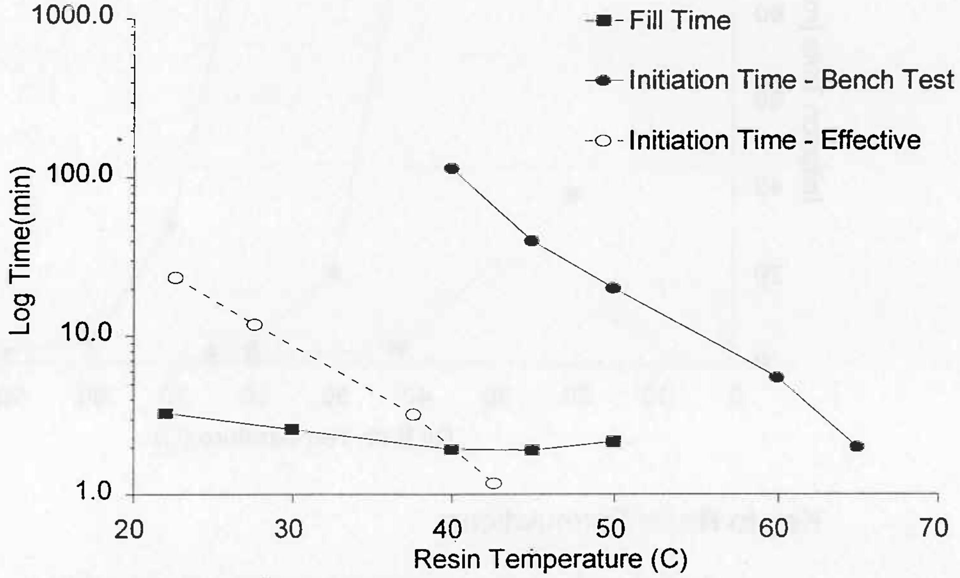


Figure 8 Relationship between fill time and initiation time

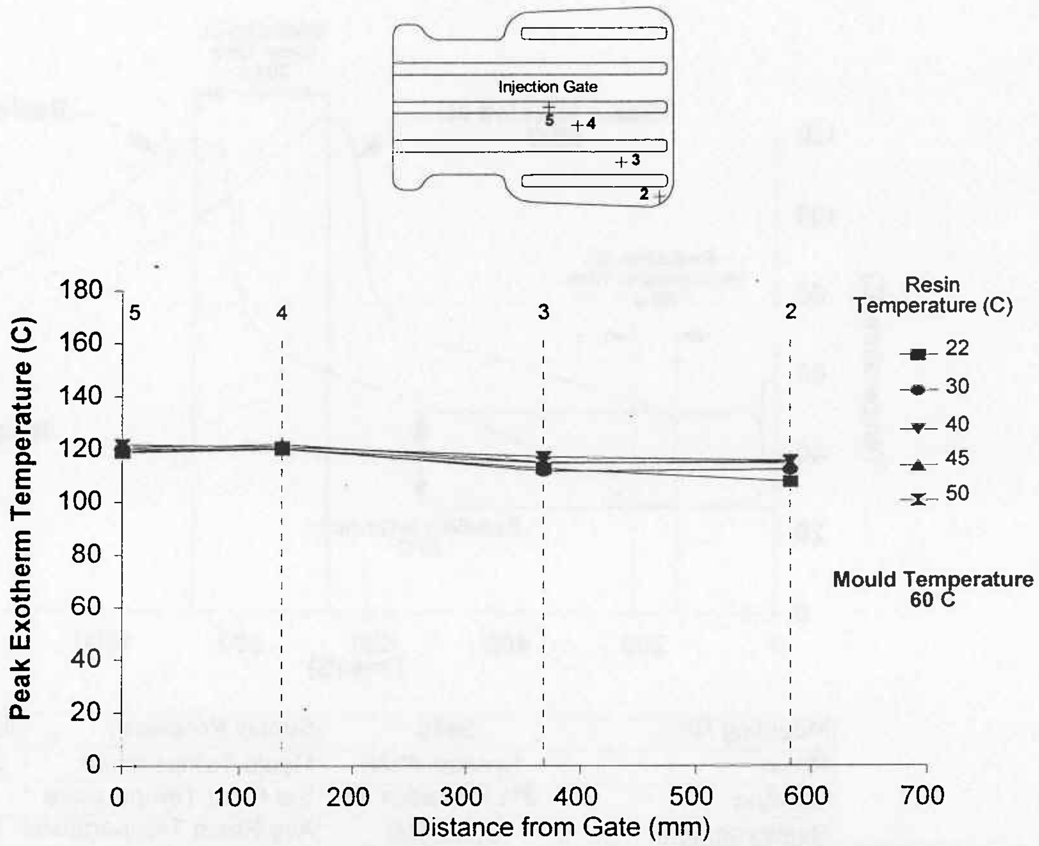
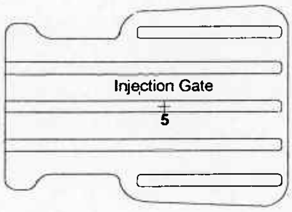
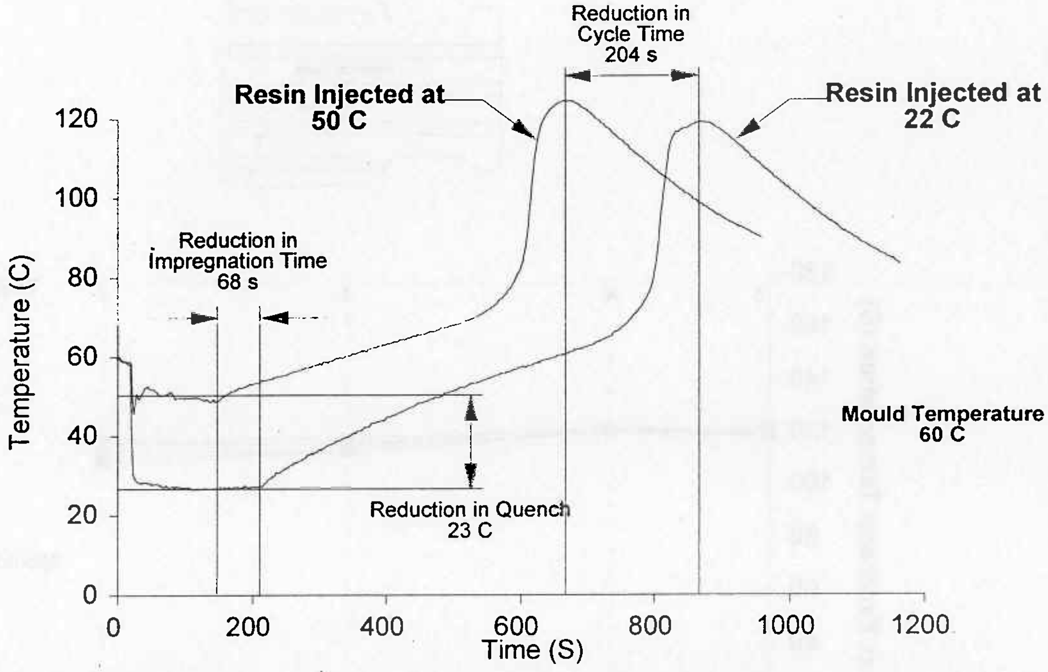


Figure 9 Effect of resin temperature on the peak exotherm temperatures across the diagonal of the undershield component





Moulding No.	5449	Supply Pressure	1.8 bar
Resin	Synolac 6345	Mould Temperature	59 C
Catalyst	2% Perkadox 16	Set Point Temperature	n/a
Reinforcement	U750-450	Avg Resin Temperature	**22 C**
Fibre Volume Fraction	16%	Power Control	n/a

Moulding No.	5446	Supply Pressure	1.8 bar
Resin	Synolac 6345	Mould Temperature	58 C
Catalyst	2% Perkadox 16	Set Point Temperature	50 C
Reinforcement	U750-450	Avg Resin Temperature	**50 C**
Fibre Volume Fraction	16%	Power Control	PID

Figure 10 Thermal histories at the injection gate to demonstrate the similarities in the resin cure kinetics between mouldings made with and without in-line microwave resin preheating

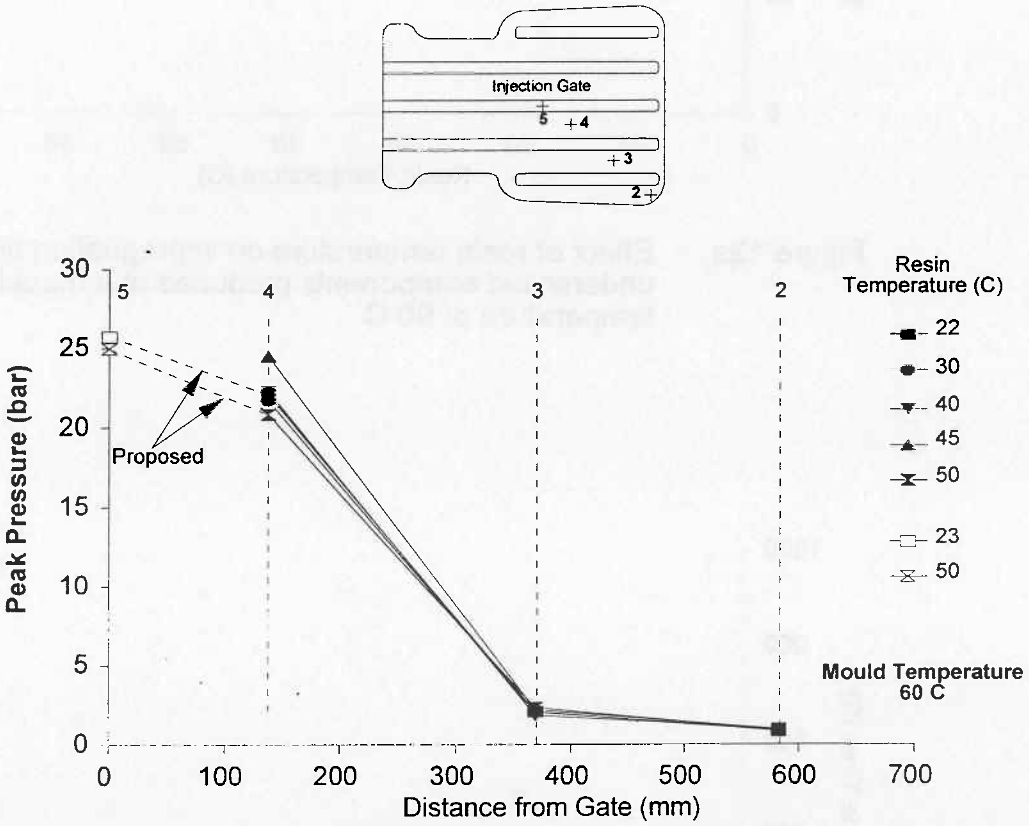


Figure 11 Effect of resin temperature on the peak pressures across the diagonal of the undershield component

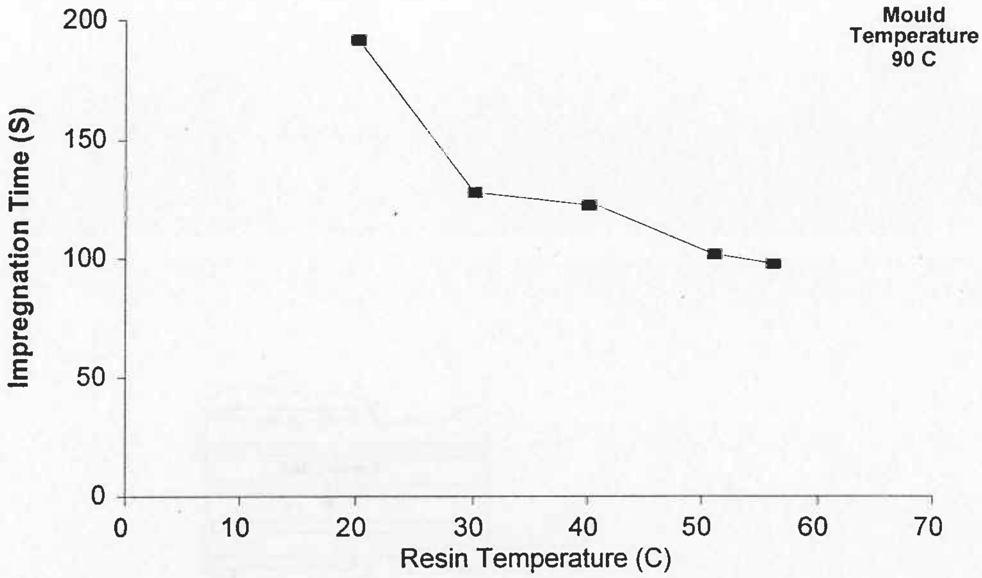


Figure 12a Effect of resin temperature on impregnation time for undershield components produced at a mould temperature of 90 C

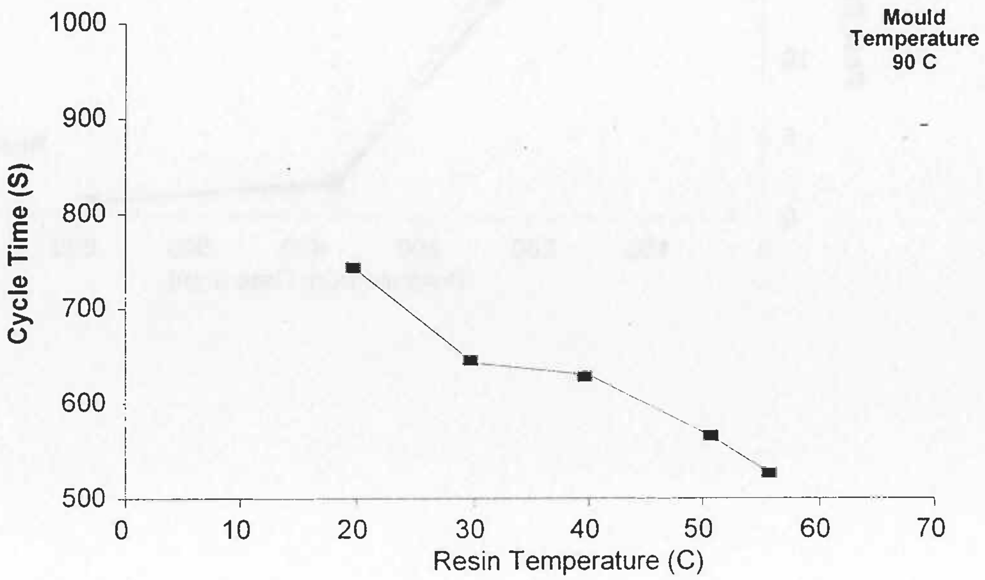


Figure 12b Effect of resin temprature on cycle time for undershield components produced at a mould temperature of 90 C

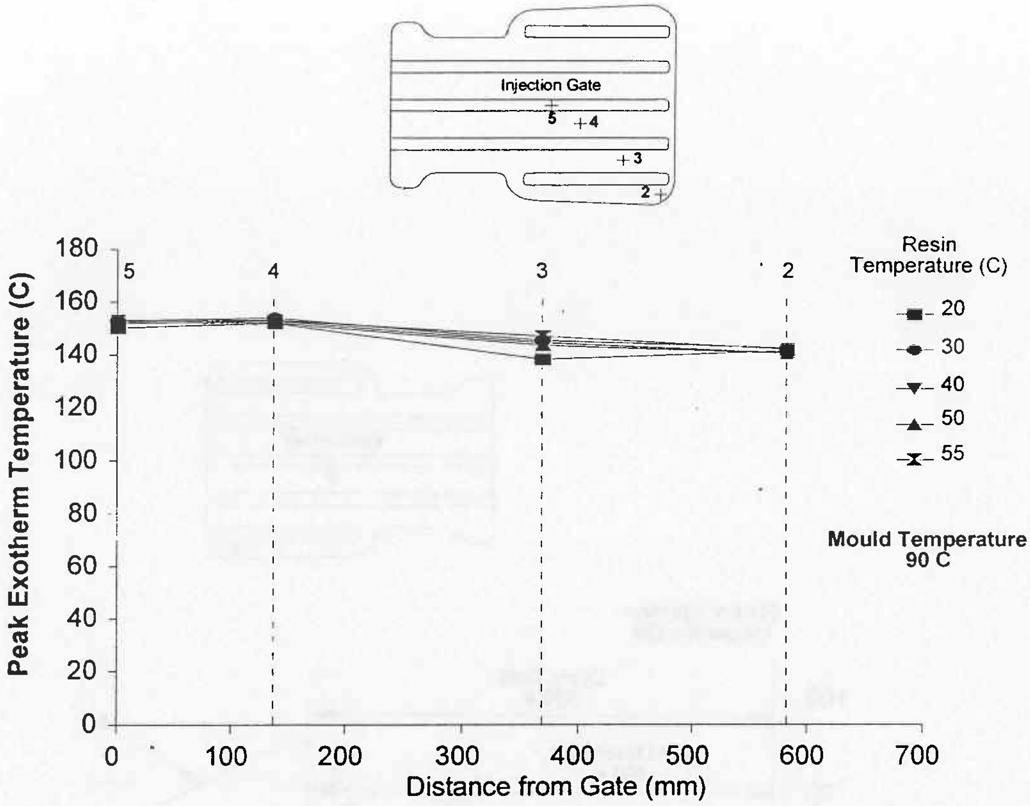


Figure 13a Effect of resin temperature on the peak exotherm temperatures across the diagonal of the undershield component

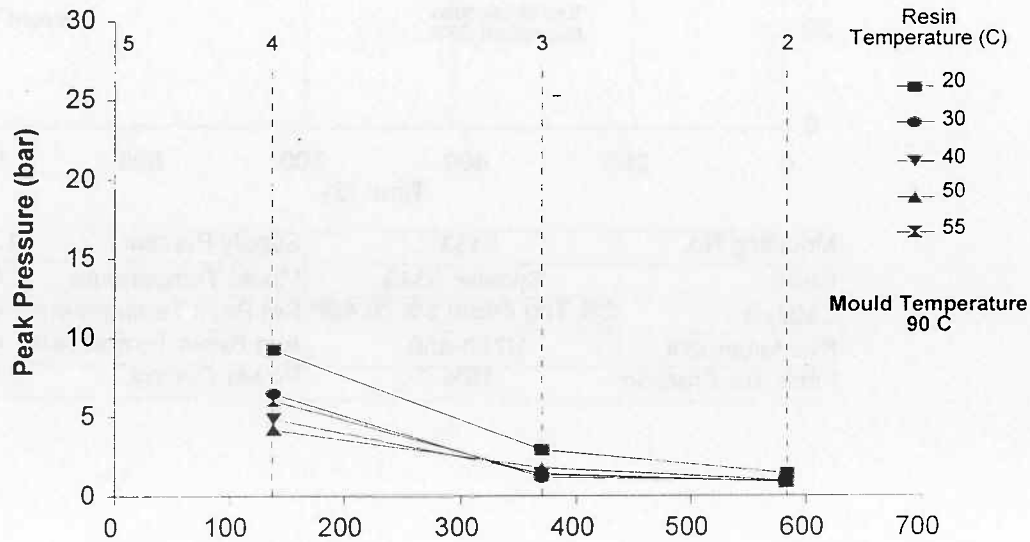
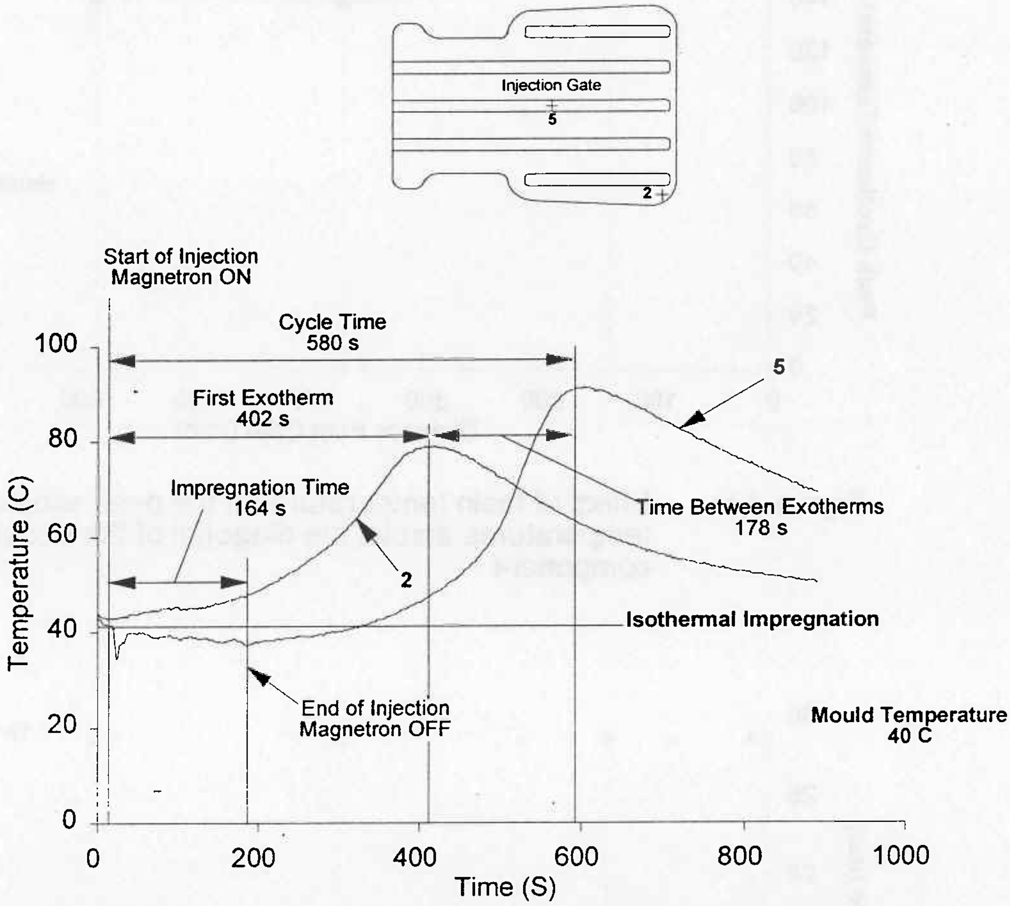


Figure 13b Effect of resin temperature on the peak pressures across the diagonal of the undershield component



Moulding No.	5433	Supply Pressure	1.8 bar
Resin	Synolac 6345	Mould Temperature	**42 C**
Catalyst	2% Trig 44B/0.5% NL49P	Set Point Temperature	40 C
Reinforcement	U750-450	Avg Resin Temperature	**40 C**
Fibre Vol. Fraction	15%	Power Control	PID

Figure 14 Thermal history at the injection gate and mould periphery for an undershield moulding produced under isothermal conditions.

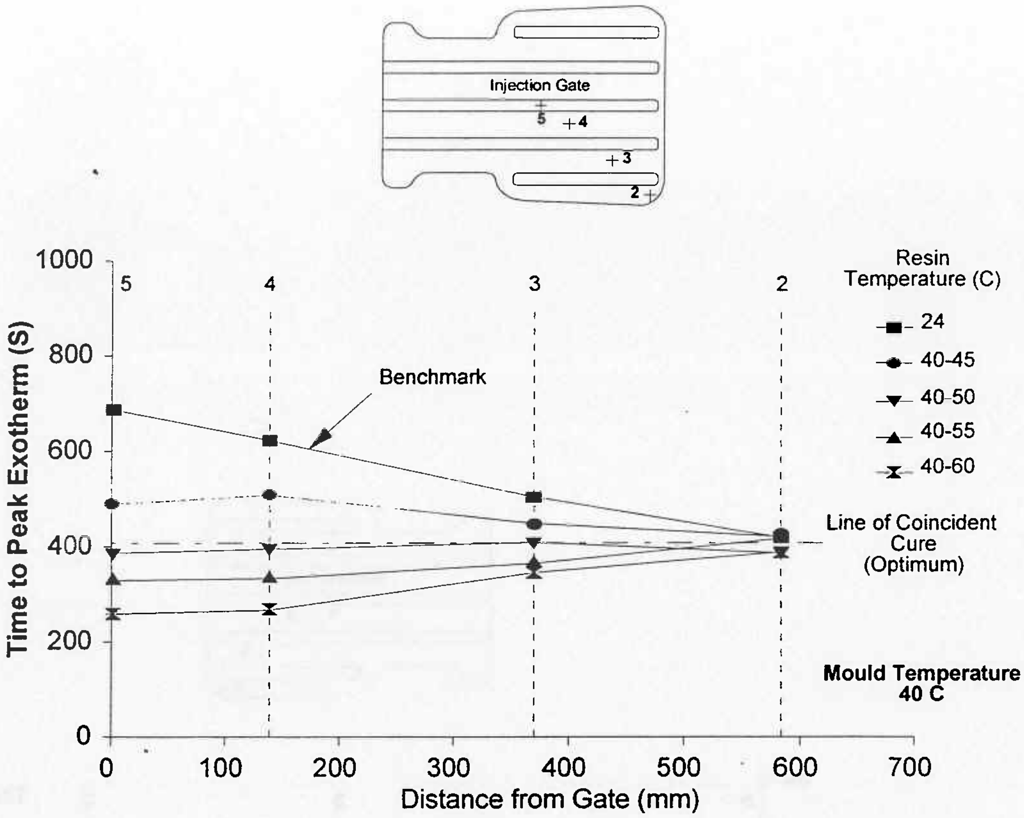


Figure 15a Effect of ramped resin temperature on the time to peak exotherm across the diagonal of the undershield component

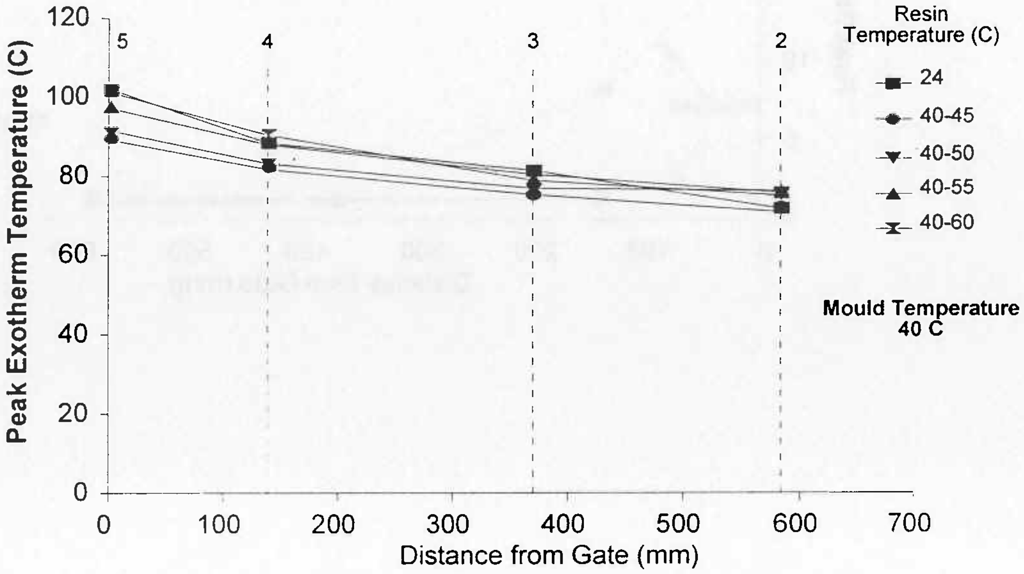


Figure 15b Effect of ramped resin temprature on the peak exotherm temperature across the diagonal of the undershield component

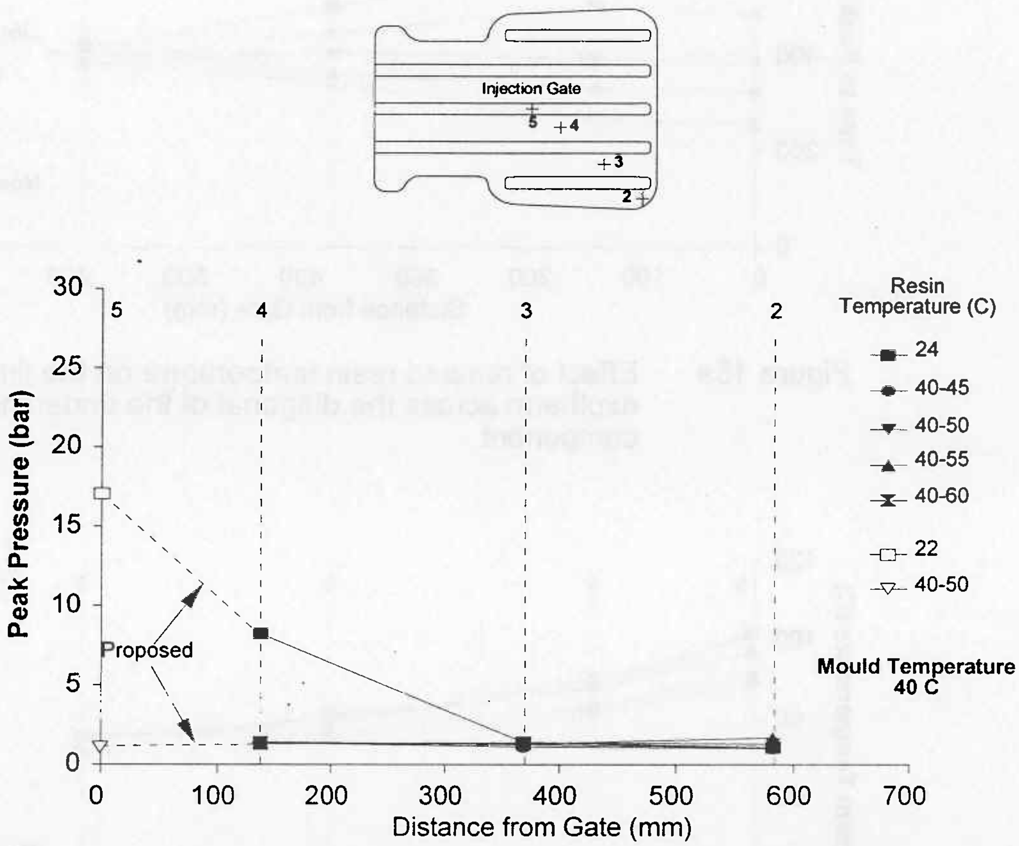
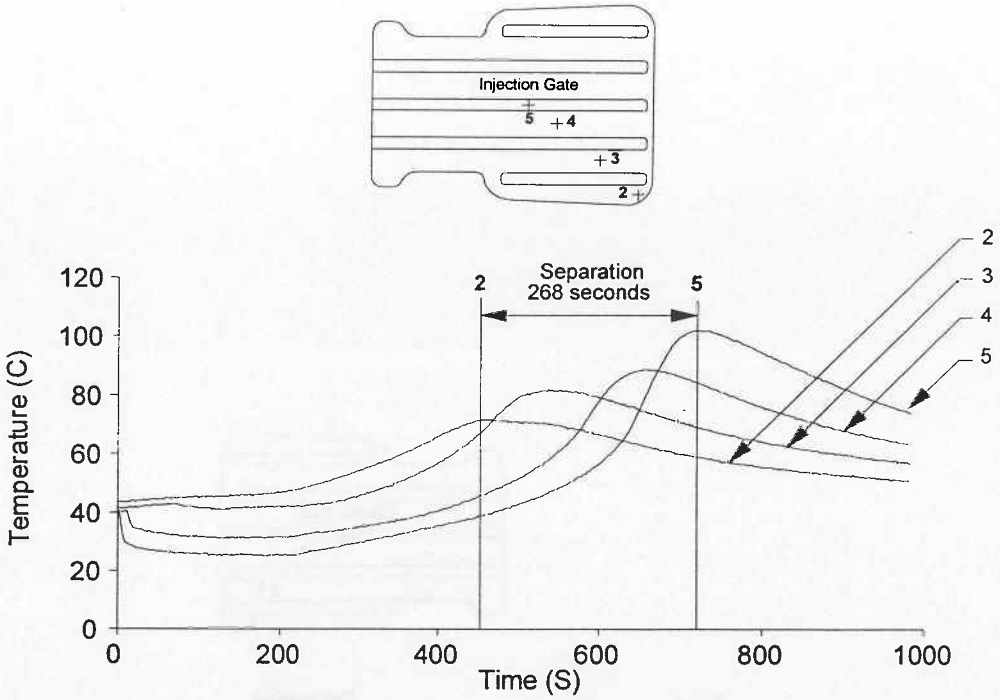
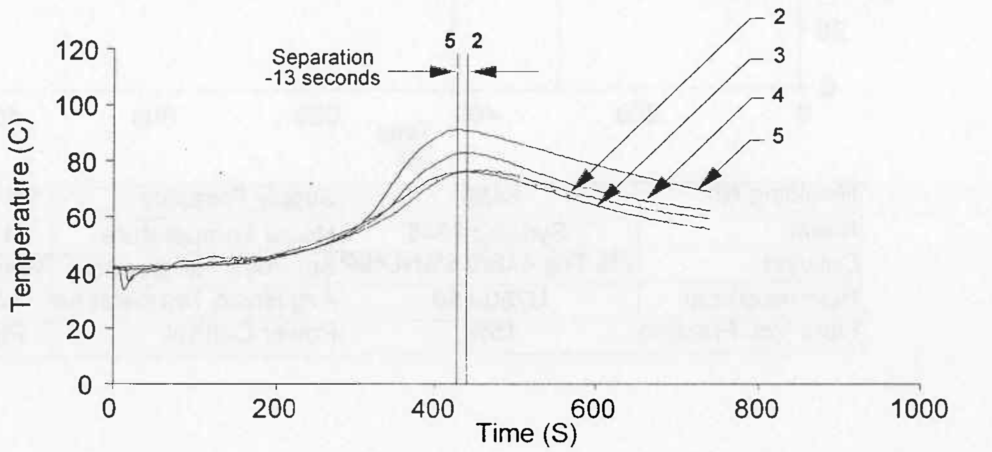


Figure 16 Effect of ramped resin temprature on the peak pressures across the diagonal of the undershield component



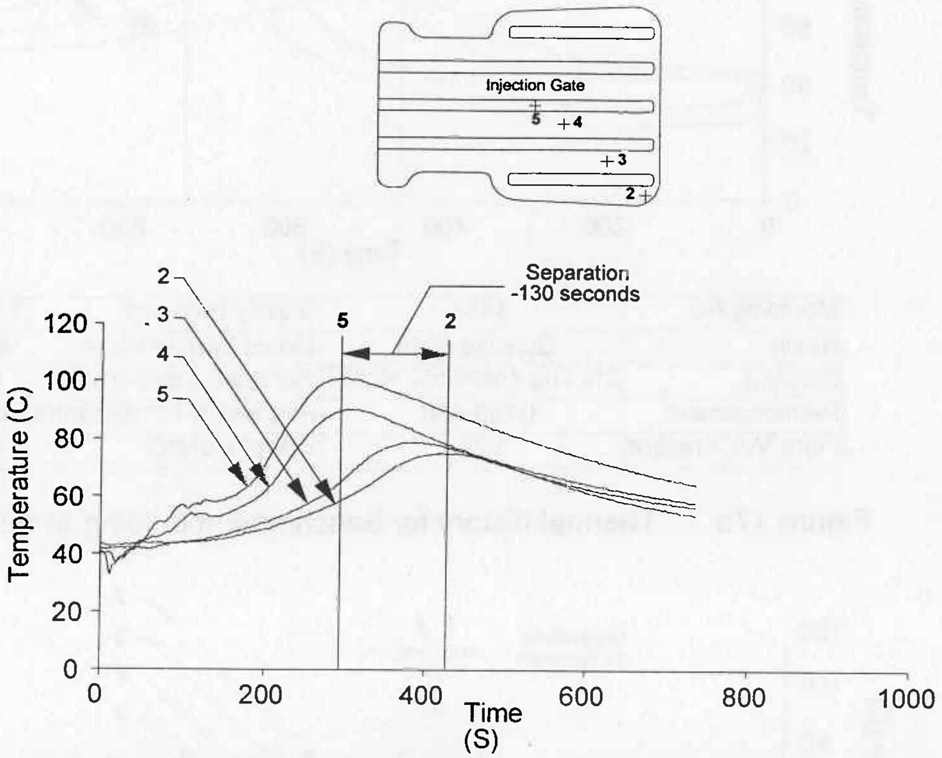
Moulding No.	5438	Supply Pressure	1.9 bar
Resin	Synolac 6345	Mould Temperature	**42 C**
Catalyst	2% Trig 44B/0.5% NL49P	Set Point Temperature	n/a
Reinforcement	U750-450	Avg Resin Temperature	**24 C**
Fibre Vol. Fraction	16%	Power Control	n/a

Figure 17a Thermal history for benchmark moulding at 40 C



Moulding No.	5434	Supply Pressure	1.9 bar
Resin	Synolac 6345	Mould Temperature	**42 C**
Catalyst	2% Trig 44B/0.5% NL49P	Set Point Temperature	**40-50C**
Reinforcement	U750-450	Avg Resin Temperature	n/a
Fibre Vol. Fraction	15%	Power Control	PID

Figure 17b Thermal history for moulding produced with the resin temperature ramped from 40-50 C during injection



Moulding No.	5435	Supply Pressure	1.9 bar
Resin	Synolac 6345	Mould Temperature	**41 C**
Catalyst	2% Trig 44B/0.5%NL49P	Set Point Temperature	**40-60 C**
Reinforcement	U750-450	Avg Resin Temperature	n/a
Fibre Vol. Fraction	15%	Power Control	PID

Figure 17c Thermal history for moulding produced with the resin temperature ramped from 40-60 C during injection