

**The consequences of rapid
processing techniques on the
physical properties of plastics
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The consequences of rapid processing techniques on the physical properties of plastics components

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ABSTRACT

During the injection moulding process, it is important to cool the moulded part as rapidly as possible to reduce cycle times and to maximise equipment utilisation. The rate at which a polymer is cooled during injection moulding will directly affect the morphology of the polymer and therefore the physical properties of the moulded part. This report gives a description of several measurement techniques used by NPL Materials Centre to measure the physical properties of amorphous, filled and unfilled semi-crystalline polymers after rapid processing. The techniques used were pressure/volume/temperature (PVT), differential scanning calorimetry (DSC), thermal conductivity (TC), tensile testing, polarised light microscopy and sink mark characterisation. In addition some mathematical modelling work has been carried out. The results presented for rapidly cooled polymers include mechanical properties, a comparison of the properties of a polymer/clay nano-composite with the virgin polymer plus crystallisation and latent heat effects. Thermal conductivity data is also reported. Polymers were cooled at rates approaching those of the injection moulding process using methods developed at the NPL Materials Centre or by injection moulding at the Wolfson Centre for Materials Processing, Brunel University. The injection moulding process used either direct cooling or pulse cooling - a recently developed cooling method designed to further reduce injection moulding cycle times. A comparison of tests on the physical properties of polymer samples moulded using the direct and pulse cooling methods is given. Also included is a study of the effect of adding thermally conducting fillers to semi-crystalline polymers to reduce injection moulding cycle times. This work provides a wide range of physical property data for polymers that have been cooled at very fast cooling rates and is more representative of commercial injection moulding than data gathered hitherto under equilibrium conditions. Pulse cooling brought about a 20% reduction in cycle time over direct cooling for the moulded polypropylene parts with no significant degradation in physical properties. Addition of nano-clay particles to nylon 6 increased the processability window of the nylon 6. Addition of undiluted aluminium filler in polypropylene achieved a 60% reduction in cycle time compared to unfilled polypropylene.

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Approved on behalf of Managing Director,
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1. INTRODUCTION

There is always a demand for reducing cycle times in the polymer injection moulding industry while maintaining or improving the level of performance of the moulded product. The physical properties of more than seven polymers with engineering applications: HDPE, PP, PET, PS, PC, 100% Konduit*, 75% Konduit in PP, 50% Konduit in PP, 25% Konduit in PP, 10% talc filled PP, 20% talc filled PP, 30% talc filled PP (all by volume percent), Nylon 6 and Nylon 6/clay nano-composite, were characterised after the polymers have been cooled rapidly either under injection moulding conditions at the Wolfson Centre for Materials Processing, Brunel University, or under conditions closely approaching those of injection moulding, using methods developed at NPL Materials Centre. This testing programme has been modified in accordance with the wishes of the customer (e-mails: from T. McCalla, 23 January 2003; to T. McCalla 28 May 2003).

The aim of this work is to determine accurate physical property data such as thermal conductivity, crystallisation, latent heat on cooling, tensile strength, Young's modulus and specific volume at ultrafast cooling rates on semi-crystalline and amorphous polymers. Due to the current design of many instruments, until now, only data obtained at or near equilibrium conditions has been available. By modifying existing techniques such as PVT and DSC, it is possible to obtain data from conditions that are closer to those experienced in injection moulding and so improve the data supplied to finite element analysis (FEA) software, and therefore the predictions, particularly where rapid processing is important.

Tensile testing was used to determine whether or not pulse cooling, an advance in injection moulding, possibly leading to reduced cycle times, will maintain or improve the physical properties of the moulded polymer, compared to the conventional, direct cooling injection moulding method. Thermal conductivity testing was used to determine whether the addition of thermally conducting fillers to polymers will also reduce cycle times and tensile testing was used to determine the effect of filler addition on the physical properties of the moulded polymer. This is the final report concluding the work carried out by NPL under contract to Brunel University as part of the EPSRC funded Enhanced Polymer Processing (EPP) research programme.

* Konduit is a masterbatch of aluminium in a polypropylene matrix. The volume fraction of aluminium in the Konduit material was determined to be 30% by thermogravimetric analysis.

2. MATERIALS

HDPE	BP Rigidex HM4560UA	NPL Code: HGH 800
HDPE	Solvay Eltex TU B 121	NPL Code: HCE 000
PET	Du Pont	NPL Code: NEH 000A
PP	Himont Moplen F30S Natural	NPL Code: HAQ 000
PP	Montell SS6100	NPL Code: NDD 000
Konduit	LNP Engineering Plastics	NPL Code: NDC 000PC
GE Plastics	Lexan 141 R	NPL Code: NDX 100 to NDZ 100; NDA 200 to NDE 200
Talc	Luznac no.2	NPL Code: NDL 200

10% Talc in PP	Brunel University	NPL Code: NDF 200
20% Talc in PP	Brunel University	NPL Code: NDG 200
30% Talc in PP	Brunel University	NPL Code: NDH 200
Nylon 6	Brunel University	NPL Code: NDI 100
Nylon 6/clay nano-composite (2% clay by volume)	Brunel University	NPL Code: NDH 100

3. EXPERIMENTAL TEST PROCEDURES

3.1 THERMAL CONDUCTIVITY (TC)

A commercial apparatus, the PVT 100 manufactured by SWO was used to measure the thermal conductivity. A schematic of the thermal conductivity equipment is shown in Figure 1. The sample was enclosed within a heated chamber, sealed at the top and bottom with PTFE seals. A probe is inserted into the molten sample and, if required, a pressure may be applied to both the probe and the sample.

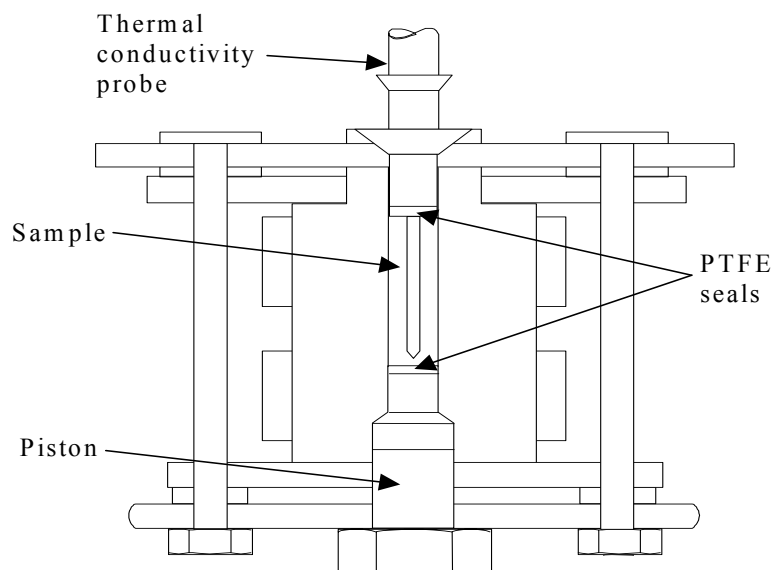


Figure 1 Diagram of thermal conductivity apparatus.

A representative curve is presented in Figure 2. Three clear regions can be observed in the curve. At short times the temperature rise is dominated by the heating of the probe, and at long times there is no further temperature rise as the response is dominated by axial losses. This equipment is based on the line source probe method; a useful review of this method has been presented by Tye [1].

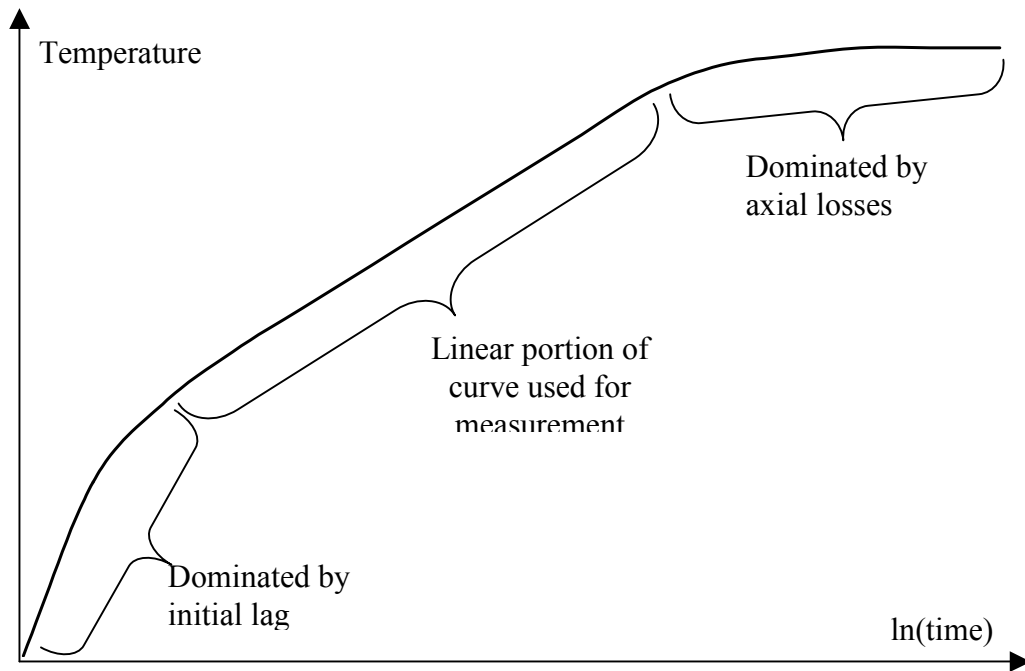


Figure 2 Schematic plot of temperature against $\ln(\text{time})$ used to calculate thermal conductivity.

The probe contains a linear heater wire and a thermocouple is located at the centre of the probe. A measurement is taken by applying a known voltage to the heater and monitoring the temperature profile by the thermocouple in the probe. To calculate the thermal conductivity the natural logarithm of the temperature rise is plotted against time. However, between these two extremes there is a region that is linear, which is representative of the sample properties. It can be shown (e.g. [1]) that the temperature rise ΔT is given by

$$\Delta T = \frac{Q}{4\pi\lambda} \ln(t).B$$

Where Q is the heat energy supplied per unit length to the sample, λ is the thermal conductivity of the sample, t is time and B is a calibration factor that accounts for deviations from the theoretical model. Hence, the gradient of the linear portion of the curve corresponds to $BQ/4\pi\lambda$, so provided the applied energy is known, it is possible to calculate the thermal conductivity of the material.

A source of uncertainty in measurements of thermal conductivity using this apparatus is the range of temperatures over which the curve is fitted to the experimental data. For glycerol at 25°C, it has been determined that variations of up to 10% can be incurred, depending on which portion of the experimental curve is used to calculate the thermal conductivity [2].

Further work is ongoing as part of the heat transfer of polymers project, MPP7.1, to make a fuller statement of the uncertainties associated with this test method.

3.2 TENSILE TESTING OF INJECTION MOULDED SAMPLES

The tensile properties of the samples have been determined using ISO standard methods. The tensile properties were measured using the ISO 527-1 1993 and ISO 527-2 1993 procedures. Tensile tests were conducted at 23 °C on ISO standard dumbbell specimens of length 170mm. Test loads were determined using a 5KN load cell.

To determine Young's modulus, axial strain was determined from the averaged outputs of 2 Instron knife-edge extensometers with a gauge length of 50 mm. The test speed was 1mm/min. Modulus data were calculated from regression of data in the 0.05% to 0.25 % strain range.

To determine tensile strength properties, axial and transverse strain were determined by video extensometer. The test speed was 100mm/min. The video extensometer data were then reprocessed to give stress/strain plots and tensile strength data was calculated from these curves.

Load cell calibration information

Equipment	UKAS accredited calibration certificate no.
Instron 4505 5KN load cell	108129



Figure 3 Moulded polypropylene part with left and right hand side tensile test bars (Wolfson Centre for Materials Processing, Brunel University).

3.3 CHARACTERISATION OF SINK MARKS OF INJECTION MOULDED SAMPLES

Shrinkage of the polymer can occur during the cooling cycle of injection moulding due to crystallisation. This is a considerable problem during the rapid processing of semi-crystalline polymers. In order to determine whether there was any difference in the shrinkage properties of a moulded polymer part when processed by pulse cooled injection moulding compared to processing by direct cooled injection moulding, a test method was developed. The method was developed using polypropylene tensile test bars and low melting point wax.

Low melting wax was poured onto one side of a pre-weighed tensile specimen bar. A flat metal panel with sharp edges was passed over the surface of the tensile test specimen, any wax sitting on the flat surface of the tensile specimen was removed, leaving only the wax that had flowed into the sink cavity remaining. The procedure was then repeated for the other side of the specimen bar. The tensile specimen bar was then re-weighed with the wax occupying the sink marks. The weight of wax retained by the sink marks gave a direct quantifiable indication of the total degree of sink for each tensile test bar. All the bars moulded under the same set of injection moulding conditions were tested and then the result averaged. The results were expressed as average quantity of wax retained per bar (g/bar) for each set of moulding conditions.

A comparison was made taking the average wax retention of ten moulded tensile test bars (five right hand side and five left hand side moulded test bars) for each mould temperature in order to compare the shrinkage properties of direct cooled and pulse cooled injection moulded test pieces.

3.4 POLARISED LIGHT MICROSCOPY

Rapid cooling of an amorphous polymer such as polycarbonate during the injection moulding process to a mould temperature below the glass transition temperature of the polymer leads to the polymer being in a 'glass-like' state, where the polymer chains are tightly packed together and molecular motion is severely restricted.

Molecular-orientation in the polymer sample can occur when the sample is 'drawn' or stretched in one direction during tensile testing or during moulding. This tends to occur more frequently in amorphous polymers below the glass transition temperature, where the polymer chains tend to have more order. The effect of 'drawing' the polymer may bring the polymer chains into a preferred direction of molecular alignment. Assuming the molecules in this situation themselves show anisotropy, the optical properties of the material will be dependent on the direction of observation.

A Leitz Wetzlar Orthoplan-Pol light microscope (NPL PB 3057) was used to investigate the structure of polycarbonate samples after tensile testing. A crossed polarizer set up was used to determine whether or not the specimens displayed anisotropy. The tensile test specimen (after stretching) is placed on a graduated rotating stage between the crossed polarizers and rotated through 360°. If anisotropy is displayed, then the intensity of the sample will change

periodically while being rotated in the path of the light. Each test specimen was examined in the region between the grips both before stretching and after stretching.

3.5 PRESSURE/VOLUME/TEMPERATURE (PVT)

Warpage and shrinkage are common problems in plastic components, due to injection mould designs being based on specific volume data obtained under equilibrium conditions. Until very recently, data for the specific volume behaviour of a polymer during rapid cooling under pressure, ie. the injection moulding process, had not been available. However, NPL have developed a PVT measurement method for measuring the PVT behaviour of polymers during rapid cooling (up to 250°C/min) and under high pressure (up to 2500 bar), Hobbs and Brown [3].

The SWO/NPL system is a direct measurement machine (Figure 4). At the start of each test the zero position for the piston must be established. To zero the displacement transducer the lower piston is screwed into the machine and two PTFE seals inserted into the cell. The linear adjustment procedure is activated from the software and the zero position is established after the piston has entered the cell and 1600 bar applied.

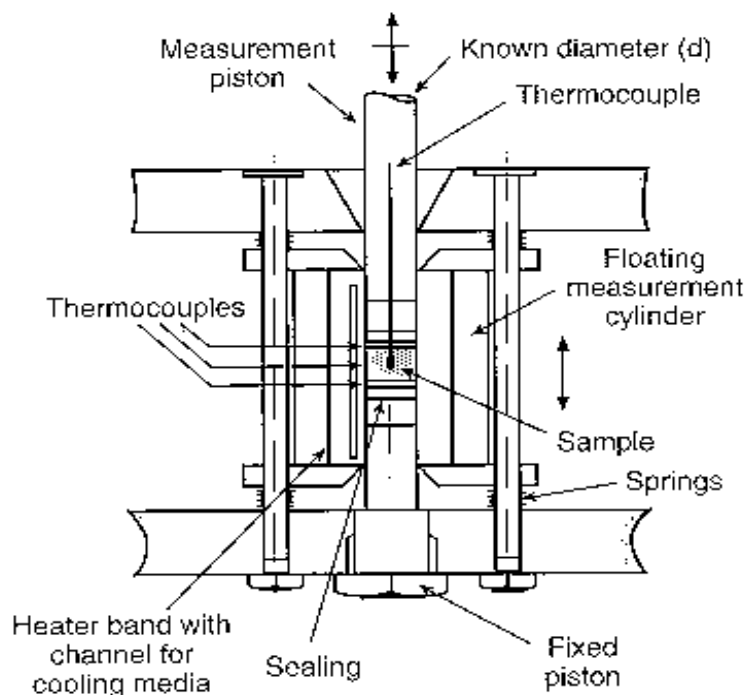


Figure 4 Diagram of pvT apparatus.

Polymer pellet or powder of a known mass is placed in the cylindrical cell and a test sample moulded in situ at 400 bar. Pressure is applied hydraulically to a piston at the top of the cell with a PTFE disc seal, for temperatures up to 250°C. An identical piston and sealing system

is located at the bottom of the cell. The polymer is heated to the initial test temperature and the chosen pressure applied through a piston. The volume is determined from the diameter and the piston position which is measured to within 0.001mm. The sample is then cooled and the changing volume measured at constant pressure. If the cooling fluid (air) is passed through liquid nitrogen, cooling rates of up to 250°C/min can be achieved. Using the final sample mass, the sample length and the cell dimensions, the software will calculate the specific volume value at each temperature. Once the specific volume vs temperature curve is obtained, the sample is reheated and the shrinkage curve for a second pressure can be measured.

The uncertainty in the specific volume stated by the manufacturer is 0.001 cc/g.

3.6 DIFFERENTIAL SCANNING CALORIMETRY (DSC)

The enthalpy of crystallisation and crystallisation temperature of the polymers were measured using a Perkin-Elmer DSC7 differential scanning calorimeter (DSC), which is a very common method for measuring the enthalpy changes during crystallisation of polymers. DSCs are based on measuring the differential power needed to maintain the sample at the same temperature as an inert reference sample. A simplified diagram of a DSC is shown in Figure 5. It consists of a sample and a reference, each with their own heaters and Pt resistance thermometers.

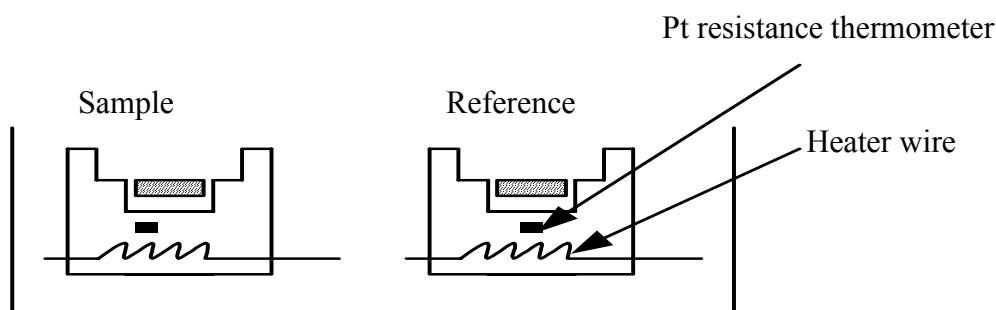


Figure 5 Schematic diagram of a DSC.

A sample of known weight is placed in the sample holder and the amount of energy, provided by an electrical heater, required to decrease the temperature of the sample per degree is measured. The test is carried out at a constant cooling rate during which the heater power is adjusted continuously and automatically to compensate for heat evolved by the sample to keep the sample holder temperature identical to that of the reference holder. The energy input from the electrical heater is measured. The instrument software will then produce a plot of heat flow vs. temperature. The minimum or maximum temperature in the heat flow curve is the crystallisation temperature and the area under this curve gives the enthalpy of crystallisation for the polymer [4].

3.7 FINITE ELEMENT ANALYSIS (FEA)

Initially two commercial FEA packages, Moldflow and ABAQUS were used to simulate the cooling of a cylinder of material. A comparison between Moldflow and experimental PVT data was made. In addition, results from an ABAQUS analysis were compared to an approximate analytical model for the determination of the temperature distribution of a cylinder during cooling.

It was found that Moldflow could be used to predict the temperature distribution in the melt and to simulate temperature-time profiles using thermal property values. However, due to limitations in the software, predictions for the temperature distribution and the temperature-time profile could not be obtained at the same cooling rate.

Heat transfer in a cylinder using constant and variable thermal property values was modelled using ABAQUS. The ABAQUS analysis showed excellent agreement with the analytical results for constant thermal property values. A discrepancy between the ABAQUS variable thermal property model and an approximate analytical model was found which is thought to be due to the approximations used in developing the analytical model. This study shows that ABAQUS is the preferred tool to model PVT behaviour.

The cooling of a cylinder of material with constant or variable properties was modelled using ABAQUS. The cylinder of material was modelled as a disc one element high. A row of 10, 8-node quadratic axisymmetric diffusive heat transfer elements (DCAX8) were used generating a disc with a radius 10 elements long. The disc was at uniform temperature initially, after which the temperature on the outside surface was reduced instantly and then maintained at a constant level. In this analysis, heat transfer takes place in the radial direction only i.e. no flux on the top and bottom surfaces of the disc. These conditions were chosen to enable a comparison to be made with an analytical model.

The analysis was conducted as a transient heat transfer analysis. Time integration in transient problems is performed with the backward Euler method in the pure conduction elements. The time increment was controlled automatically by ABAQUS. In transient heat transfer analysis with second-order elements there is a relationship between the minimum usable time increment and the element size. The effect of changing both this minimum time increment and maximum time increment was investigated. In both cases, increases in the time increments caused the predicted time-temperature plots to become less smooth. Hence the calculated minimum time increment and a small maximum time increment were chosen for the analysis. Even with small time increments the analysis was completed quickly, with a CPU time of 15.53 seconds.

4. EXPERIMENTAL RESULTS AND DISCUSSION

4.1 THERMAL CONDUCTIVITY OF POLYMERS

4.1.1. High Density Polyethylene (HDPE)

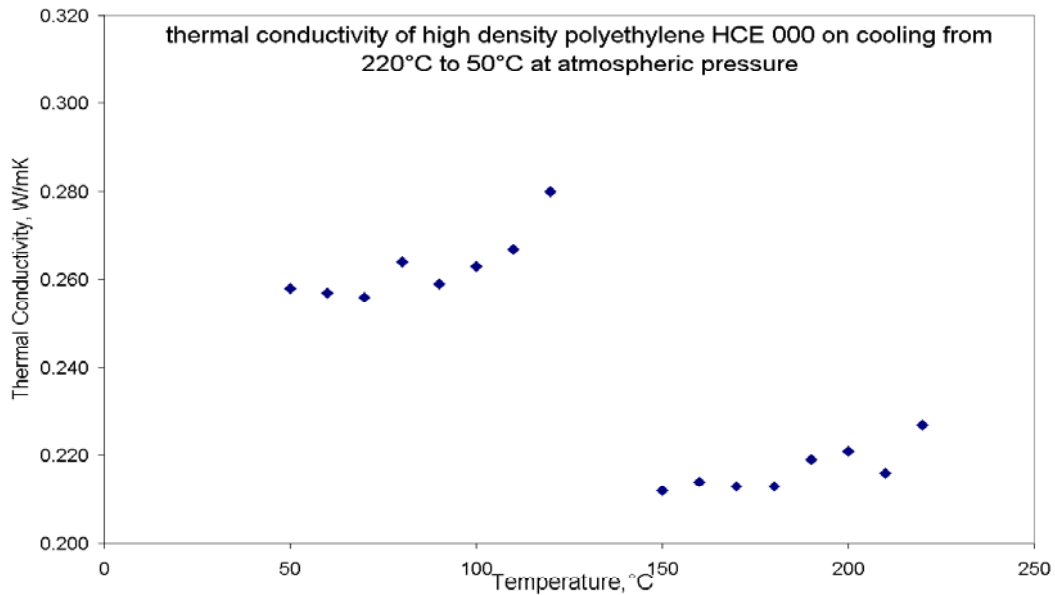


Figure 6 Thermal conductivity of high density polythene on cooling from 220°C to 50°C at atmospheric pressure.

4.1.2. Polypropylene (PP)

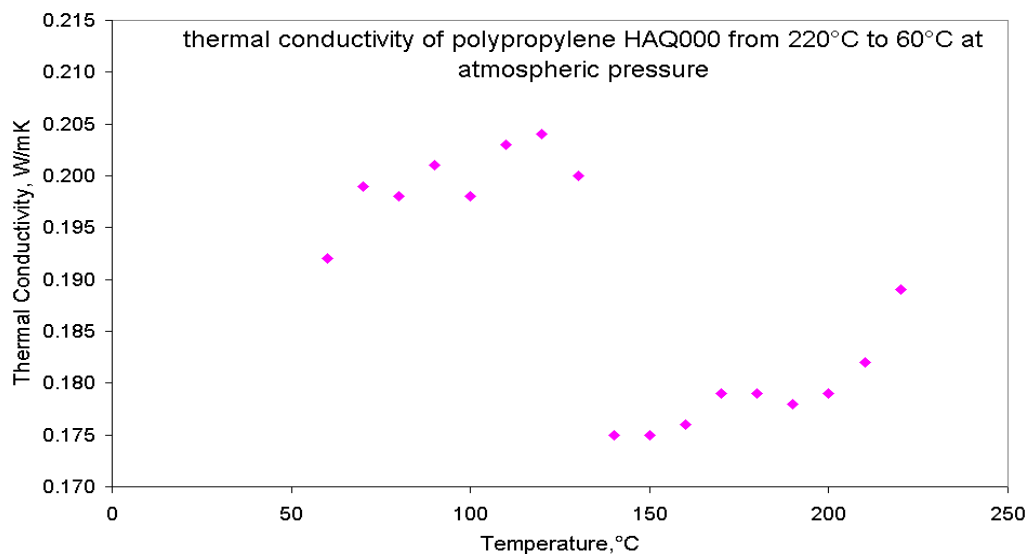


Figure 7 Thermal conductivity of polypropylene on cooling from 220°C to 60°C at atmospheric pressure.

4.1.3. Poly(ethylene terephthalate) (PET)

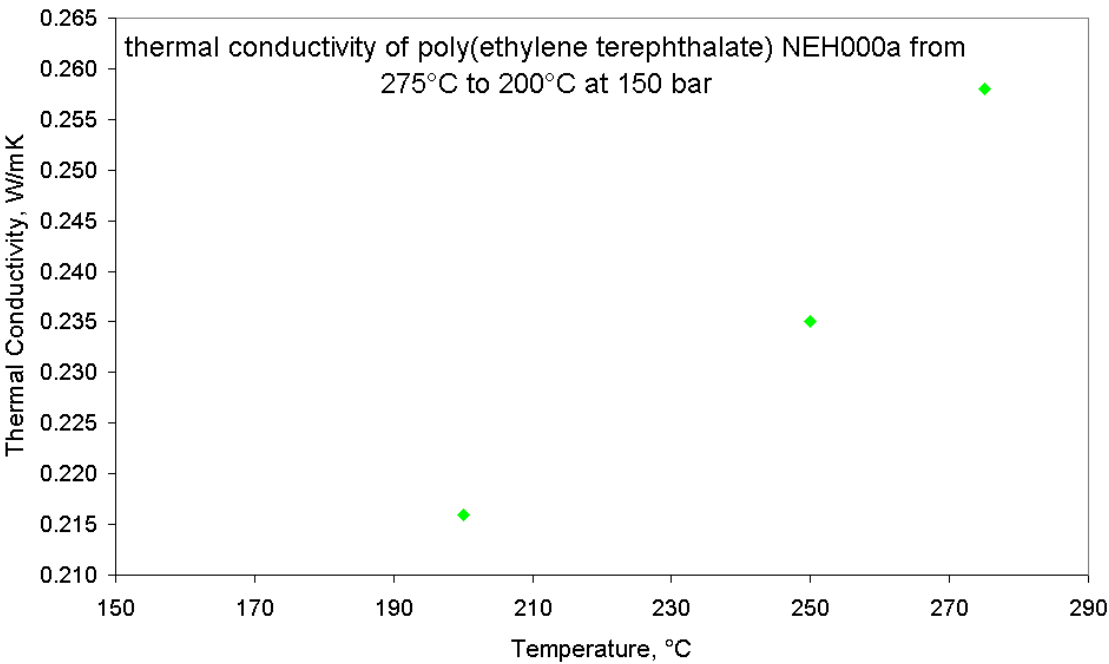


Figure 8 Thermal conductivity of poly(ethylene terephthalate) from 275°C to 200°C at 150 bar.

4.1.4 Polystyrene (PS)

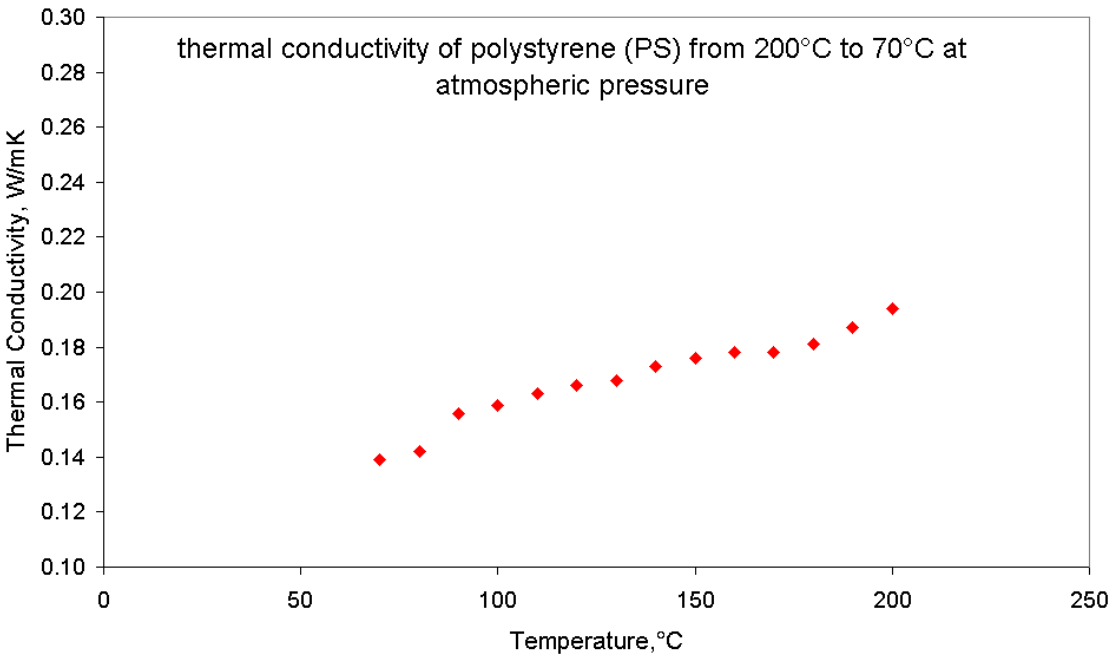


Figure 9 Thermal conductivity of polystyrene from 200°C to 70°C at atmospheric pressure.

Figures 6 and 7 show the thermal conductivity of two semi-crystalline polymers during cooling. The thermal conductivity of both HDPE and PP increases with decreasing temperature, through the solidification region. This is due to the semi-crystalline polymer adopting a regular crystalline arrangement during solidification. In this arrangement, the close packing of the polymer chains allows easier transfer of heat than in the looser, more random arrangement of the polymer chains in the melt state.

Figures 8 and 9 show the thermal conductivity of two amorphous polymers during cooling. The thermal conductivity of both PET and PS decreases with decreasing temperature. For PS on cooling, a relatively sharp decrease in thermal conductivity values occurs at the glass transition temperature between 100 C and 90 C.

Thermal conductivity data from the SWO pvT instrument obtained under these conditions and under pressure can be used to model heat transfer properties of polymers under polymer processing conditions.

4.2 COMPARISON OF PROPERTIES OF PULSE COOLED AND DIRECT COOLED FILLED AND UNFILLED INJECTION MOULDED POLYMERS

4.2.1 Polypropylene

4.2.1.a) mechanical properties

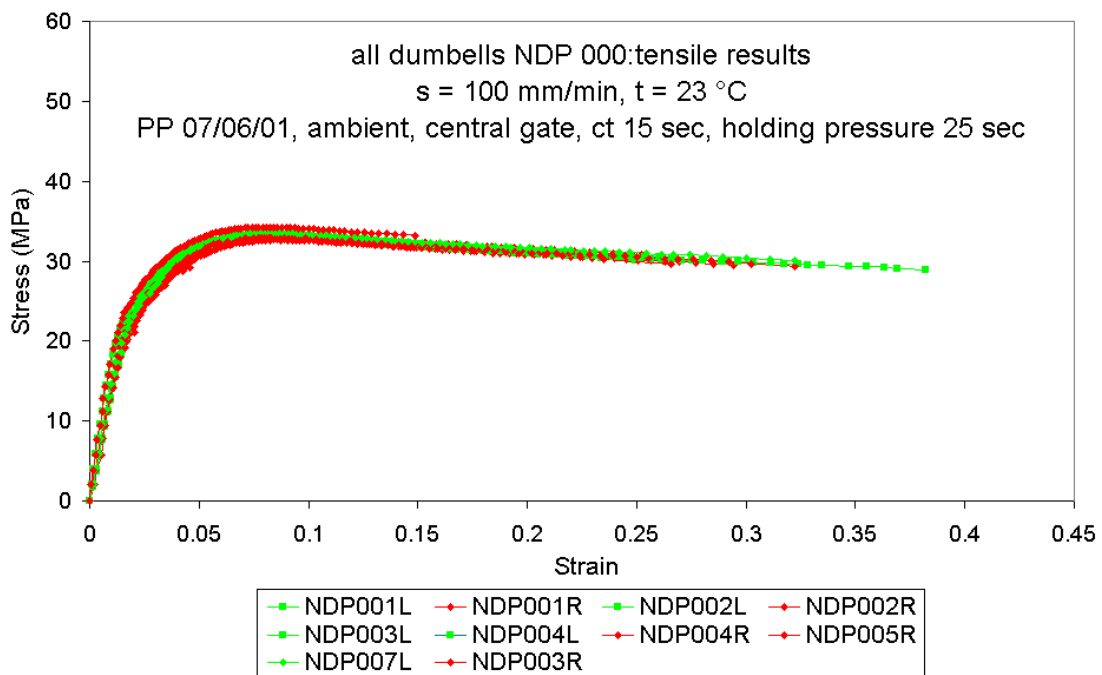


Figure 10 Comparison of tensile properties of left hand and right hand side polypropylene test bars moulded at ambient temperature using conventional (direct) cooling.

PP was injection moulded in the tool previously described, held at ambient temperature and cooled using direct cooling. The mechanical properties of five left side and five right side tensile test bars were compared. By comparing maximum stress and stress at failure (Figure 10), no significant difference in the mechanical behaviours of the left and right test bars was

found. Therefore subsequent test work looks primarily at the properties of the left hand test bars.

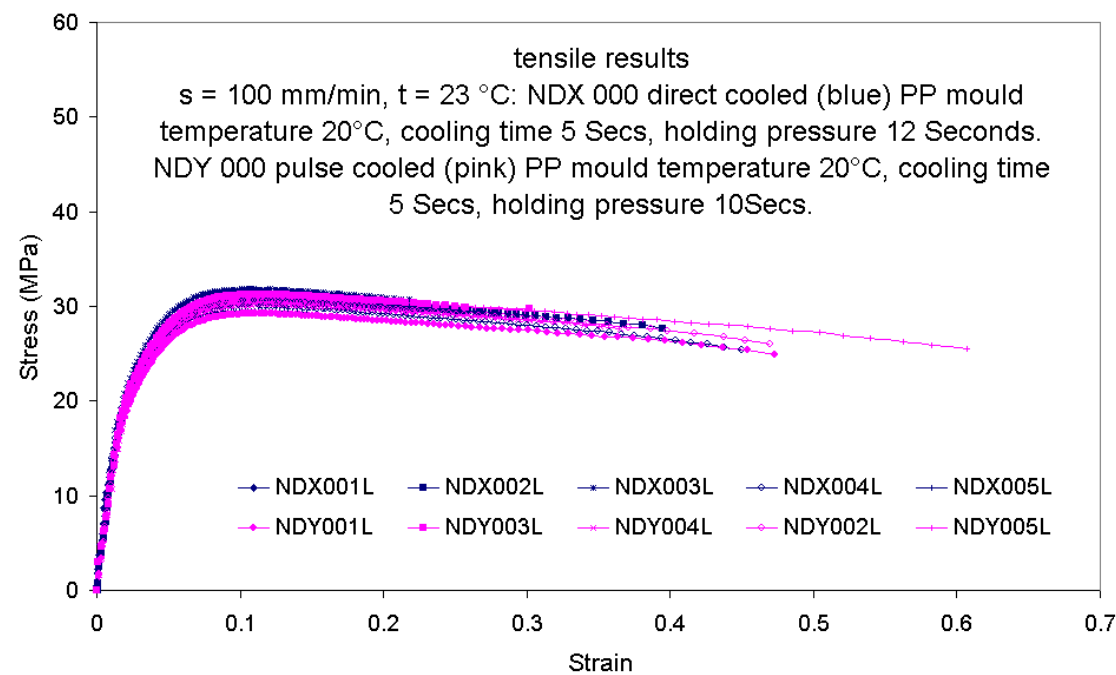


Figure 11 Comparison of tensile properties of polypropylene test bars moulded at 20°C using direct cooling and pulse cooling methods.

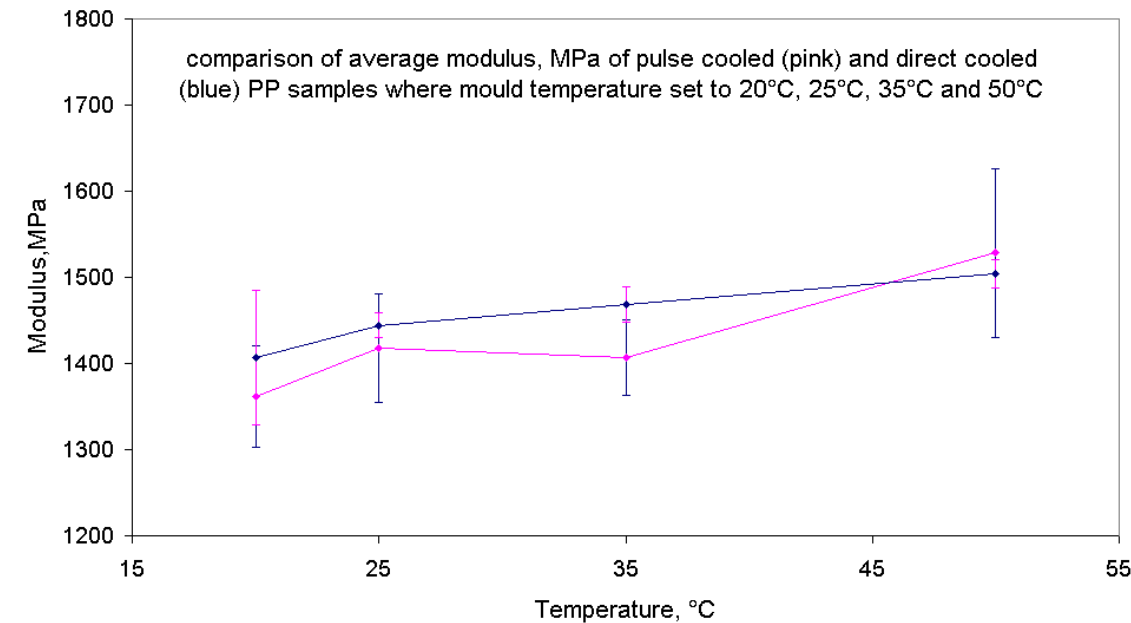


Figure 12 Comparison of the average modulus of polypropylene test bars moulded at 20°C, 25°C, 35°C and 50°C using direct cooling and pulse cooling methods.

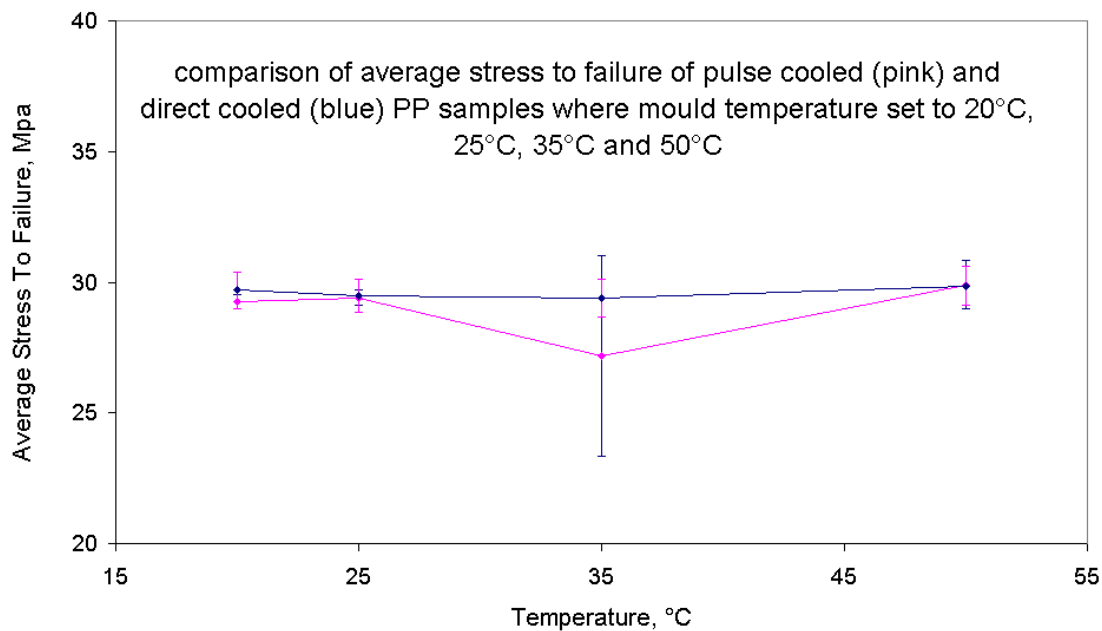


Figure 13 Comparison of the average stress to failure of polypropylene test bars moulded at 20°C, 25°C, 35°C and 50°C using direct cooling and pulse cooling methods.

The test bars were moulded with mould temperatures set at 20°C, 25°C, 35°C and 50°C. The mechanical properties of five left hand side direct cooled and five left hand side pulse cooled tensile test bars moulded at each temperature were compared. A comparison of the stress/strain curve (Figure 11) for pulse and direct cooled PP at each mould temperature produced no significant difference in the average stress to failure at each mould temperature for the two cooling methods (Figure 13), the error bars represent the standard deviation of the five test specimens. Comparison of modulus of the pulse and direct cooled PP at each mould temperature (Figure 12) also produced no significant difference in the values of the modulus of PP cooled by the two different methods (the error bars represent the standard deviation of the five test specimens). Therefore from comparing the modulus and stress at failure properties of direct and pulse cooled PP moulded over a range of mould temperatures, the mechanical behaviour for PP cooled by the two different methods was found to be similar.

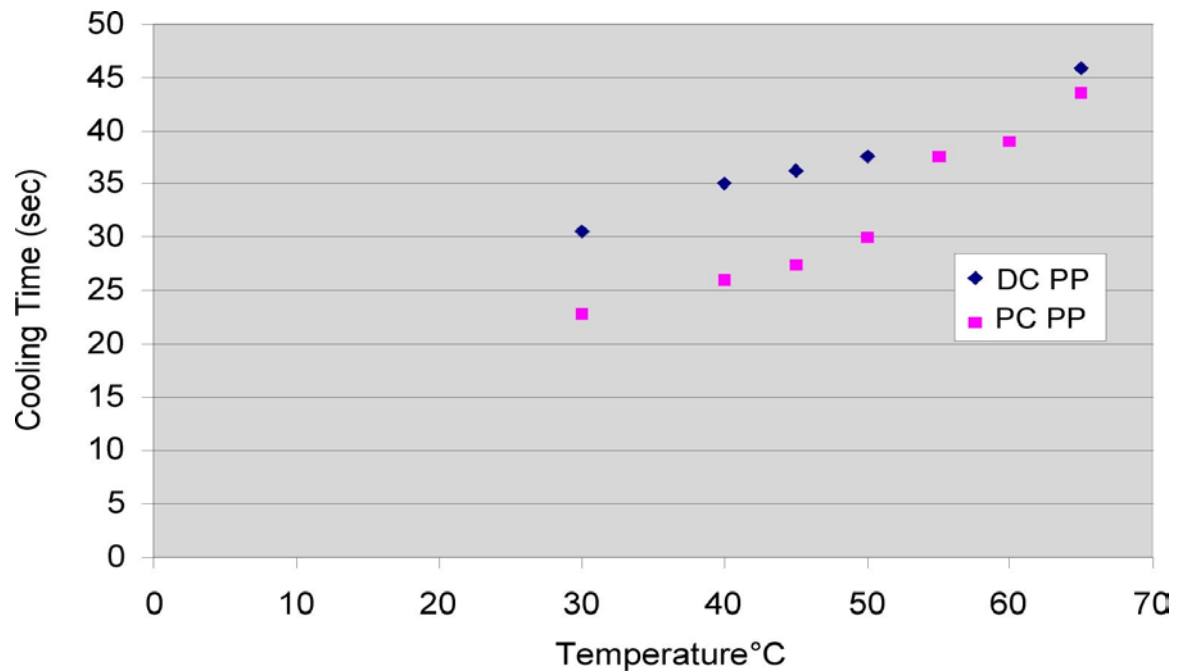


Figure 14 Cooling time vs. temperature profile for polypropylene moulded over a range of mould temperatures between 65°C and 30°C.

For lower mould temperatures, pulse cooling offers a 20% reduction in cycle times compared to direct cooling, for PP [5] (Figure 14). Results provided by Wolfson Centre for Materials Processing, Brunel University.

4.2.1.b) Sink Marks

The tests were carried out on ten (five right hand side and five left hand side) moulded polypropylene tensile test bars for each mould temperature for both pulse and direct cooled samples. The average quantity of wax retained per bar was calculated and used as a measure of degree of shrinkage of the samples, see Table 1.

Wax Retention Properties of Pulse and Direct Cooled Injection Moulded PP Samples					
Pulse Cooled			Direct Cooled		
Sample	Mould Temperature, °C	Average wax retention g/bar	Sample	Mould Temperature, °C	Average wax retention g/bar
NDY 000	20	0.1920	NDX 000	20	0.2083
NDZ 000	25	0.3782	NDC 100	25	0.3141
NDA 100	35	0.2188	NDD 100	35	0.3302
NDB 100	50	0.2513	NDE 100	50	0.3506

Table 1 Comparison of wax retention properties of pulse and direct cooled polypropylene test bars moulded at 20°C, 25°C, 35°C and 50°C.

A higher degree of shrinkage was found for samples processed at higher mould temperatures for both direct and pulse cooling conditions. There also appears to be a tendency for the wax retention of the pulse cooled samples to be lower than for the direct cooled samples, indicating that the degree of shrinkage across the range of mould temperatures is less for the pulse cooled than for the direct cooled polypropylene moulded test pieces. The pulse cooled samples moulded at a mould temperature of 25°C do not follow these two trends due to four samples with very high wax retention values increasing the average wax weight retained per bar.

4.2.2 Konduit Filled Polypropylene

To try to improve the heat transfer properties of the polypropylene, a commercially available conductive aluminium filled polymer was added to the polypropylene. There are a number of reports in the literature regarding metal fillers [6-9]. The time required to cool a moulded part is the limiting step in many processing routes. Hence, there is a strong connection between productivity and thermal conductivity.

A series of five materials was produced, containing various proportions of Konduit and unfilled PP (Montell SS610). The Konduit material consisted of aluminium in a PP matrix. Batches were produced with 0%, 25%, 50%, 75% and 100% of Konduit being filled by volume, with the remainder being unfilled PP. The materials were compounded at the Wolfson Centre for Materials Processing using a Betol twin-screw extruder and then pelletised.

4.2.2.a) mechanical properties

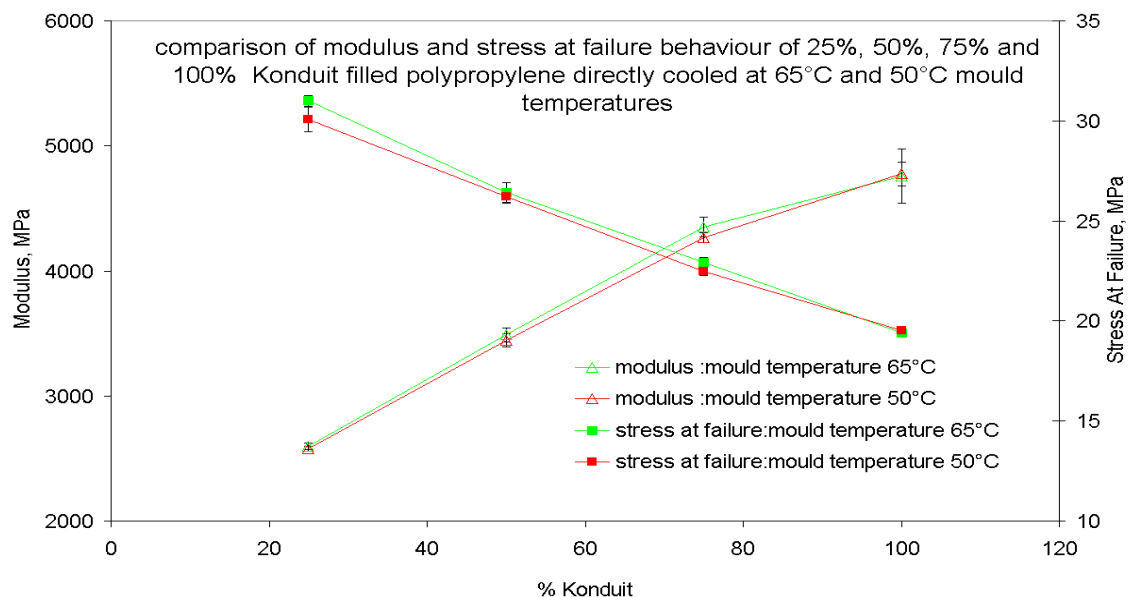


Figure 15 Comparison of modulus and stress at failure behaviour of 25%, 50%, 75% and 100% Konduit filled polypropylene directly cooled at 65°C and 50°C mould temperatures.

Test pieces from four different mix ratios of Konduit to PP were injection moulded with the mould being cooled by direct cooling. The percentages of Konduit blended with PP were 25%, 50%, 75% and 100% Konduit respectively. The test bars were moulded with mould temperatures set at 50°C and 65°C. The mechanical properties of five left hand side direct cooled tensile test bars for each of the four mix ratios moulded at each of the two mould temperatures were compared. A comparison of the average stress to failure and modulus for the four different Konduit: PP mix ratios moulded at 65°C and 50°C respectively (Figure 15) did not result in a significant difference in the values of the two properties (the error bars represent the standard deviation of the five test specimens). Therefore from comparing the modulus and stress at failure properties of direct cooled Konduit to PP blends of different mix ratios injection moulded at mould temperatures of 65°C and 50°C, mould temperature did not produce a significant difference in the mechanical behaviour of the materials.

As expected the modulus increased with % Konduit and the stress at failure reduced.

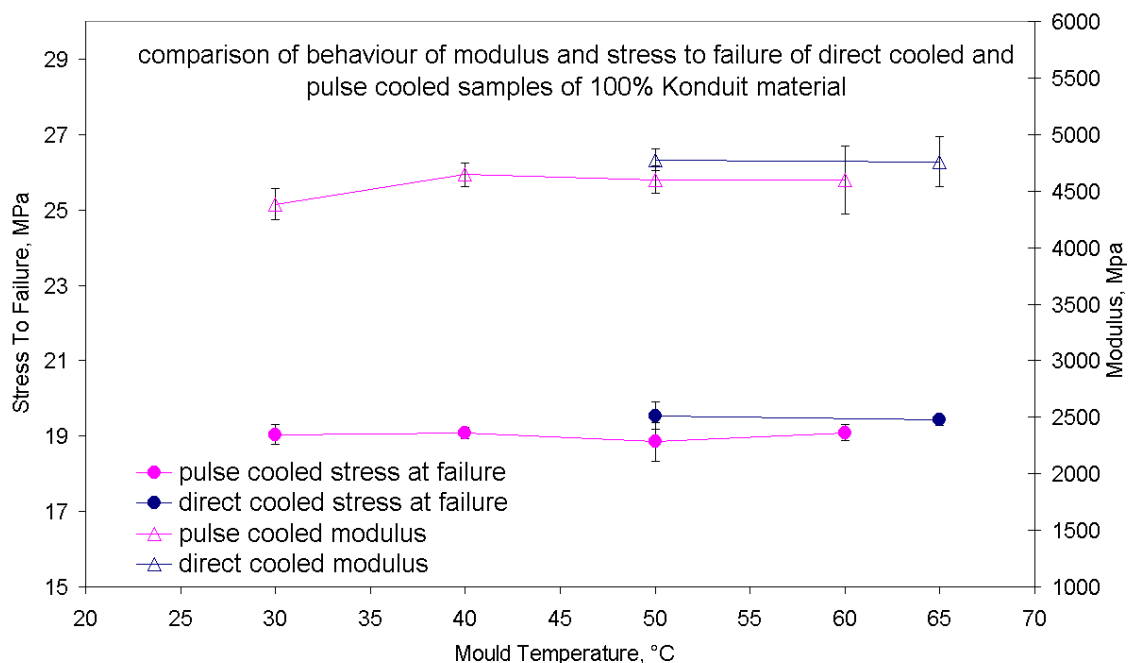


Figure 16 Comparison of modulus and stress to failure of direct cooled and pulse cooled 100% Konduit test bars moulded over a range of temperatures from 65°C to 30°C.

A comparison using 100% Konduit injection moulded into test pieces was undertaken to determine whether or not the cooling method influenced the mechanical properties of the material. The direct cooled test bars were moulded with mould temperatures set at 50°C and 65°C. The pulse cooled test bars were moulded with mould temperatures set at 60°C, 50°C, 40°C and 30°C. The mechanical properties of direct cooled and pulse cooled tensile test bars moulded at each temperature were tested. A comparison of the modulus at 50°C mould temperature for the two cooling methods (Figure 16) produced no significant difference (the error bars represent the standard deviation of the eight test specimens). A comparison of stress at failure of the pulse and direct cooled undiluted Konduit at each mould temperature produced no significant difference at a 95% confidence level (the error bars represent two standard deviations) in the values of the stress at failure of Konduit cooled by the two different methods.

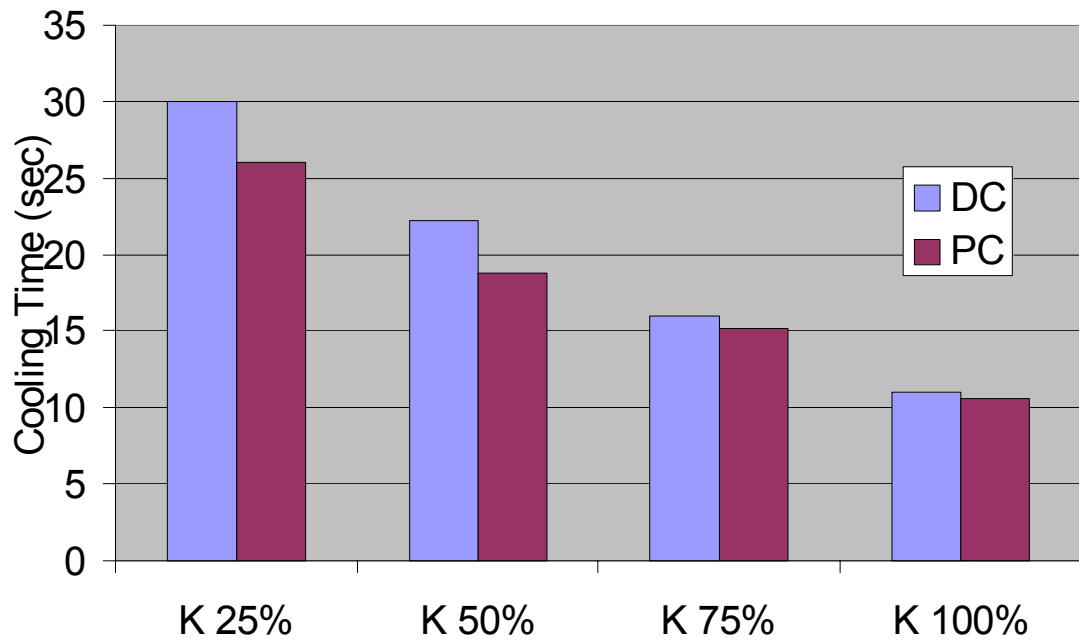


Figure 17 Comparison of direct and pulse cooling times for 25%, 50%, 75% and 100% Konduit filled polypropylene at 50°C mould temperature.

The application of pulse cooling results in a decrease in cooling time compared to direct cooling for all fill levels of Konduit in PP tested and for all mould temperatures investigated (Figure 17). Results provided by Wolfson Centre for Materials Processing, Brunel University [5].

4.2.2.b) thermal conductivity

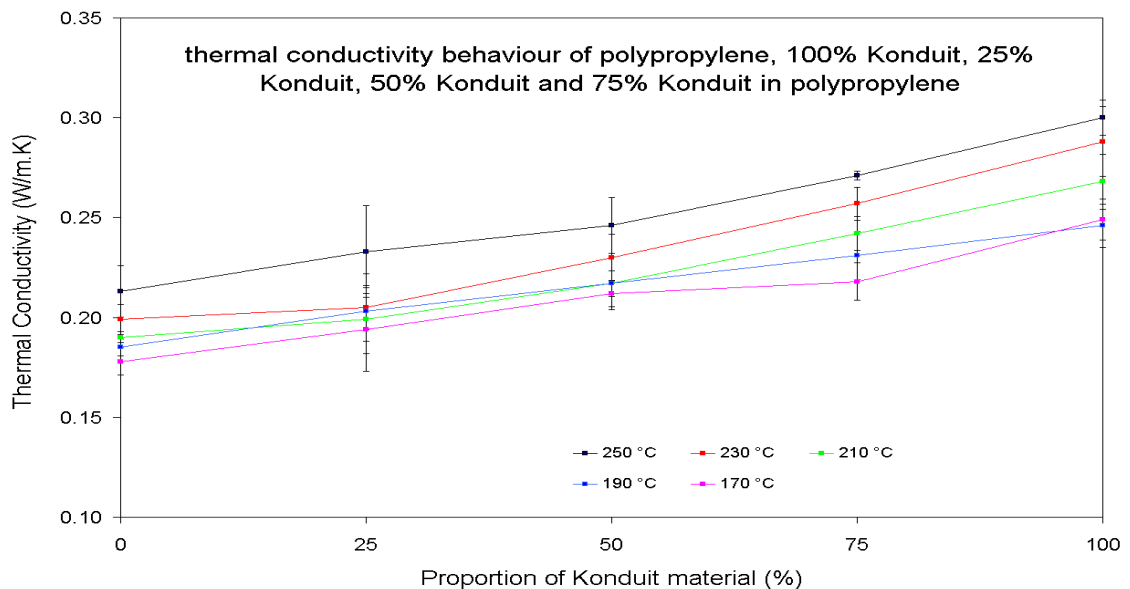


Figure 18 Comparison of the thermal conductivity behaviour of 25%, 50%, 75% and 100% Konduit filled polypropylene at 250°C, 230°C, 210°C, 190°C and 170°C and 150 Bar pressure.

Measurements were taken at five temperatures (170°C, 190°C, 210°C, 230°C and 250°C) for each of the five materials under 150 bar pressure. It can be seen that the thermal conductivity increases with increasing proportion of Konduit material. The error bars represent the standard deviation of five measurements taken at each temperature for each material. There is a relatively clear concave trend corresponding to greater improvements in thermal conductivity at higher volume fractions of Konduit material. An increase in thermal conductivity with temperature can also be seen.

4.2.3. Talc Filled Polypropylene

To try to improve the heat transfer properties of the polypropylene, a commercially available conductive talc filler was added to the polypropylene.

A series of three materials was produced, containing various proportions of talc and unfilled PP (Montell SS610). Materials were produced with 10%, 20% and 30% of talc being filled by volume, with the remainder being unfilled PP. The materials were compounded at the Wolfson Centre for Materials Processing using a Betol twin-screw extruder and then pelletised.

4.2.3.a) Thermal Conductivity

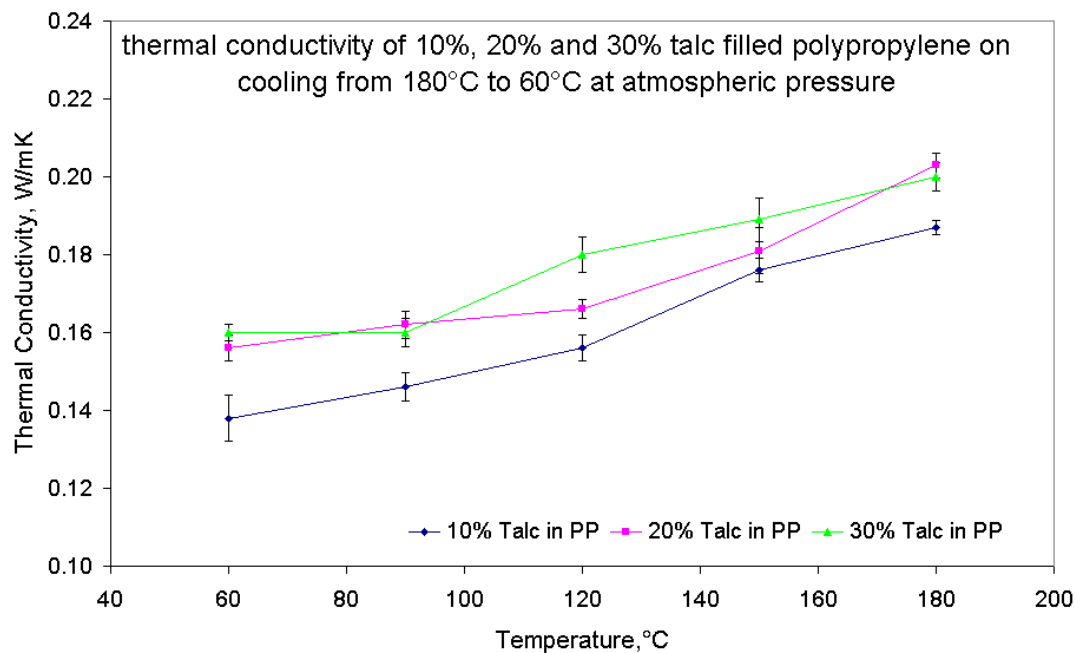


Figure 19 Thermal conductivity of 10%, 20% and 30% talc filled polypropylene on cooling from 180°C to 60°C at atmospheric pressure.

Measurements were taken at five temperatures (180°C, 150°C, 120°C, 90°C and 60°C) for each of the three materials. It can be seen that generally the thermal conductivity increases with increasing proportion of talc fill level. However, at 180°C and 90°C the thermal conductivity of the 30% talc material is lower than that of the 20% talc containing material. The difference is within one standard deviation for the five test results at the two temperatures and so is not significant. The error bars represent the standard deviation of five

measurements taken at each temperature for each material. An increase in thermal conductivity with temperature is also seen as with Konduit filled polypropylene.

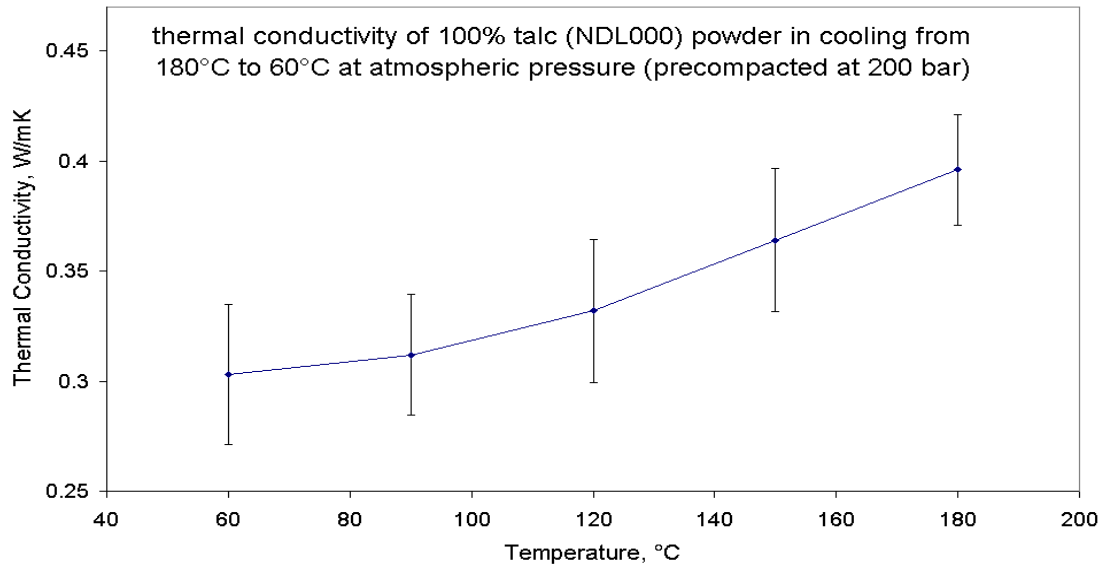


Figure 20 Thermal conductivity of 100% talc powder, precompacted at 200 bar, in cooling from 180°C to 60°C at atmospheric pressure.

The thermal conductivity of talc powder was measured from 180°C to 60°C after pre-compaction at 200 bar. The thermal conductivity of the talc sample decreases with decreasing temperature. The thermal conductivity values are lower than would be expected for solid talc because of the entrapment of air within the sample due to the sample being in powder format. The large standard deviation for the measured five points for the sample at each temperature could also be attributed to the entrapment of air within the powder sample.

4.2.4 Polycarbonate (PC)

4.2.4.a) mechanical properties

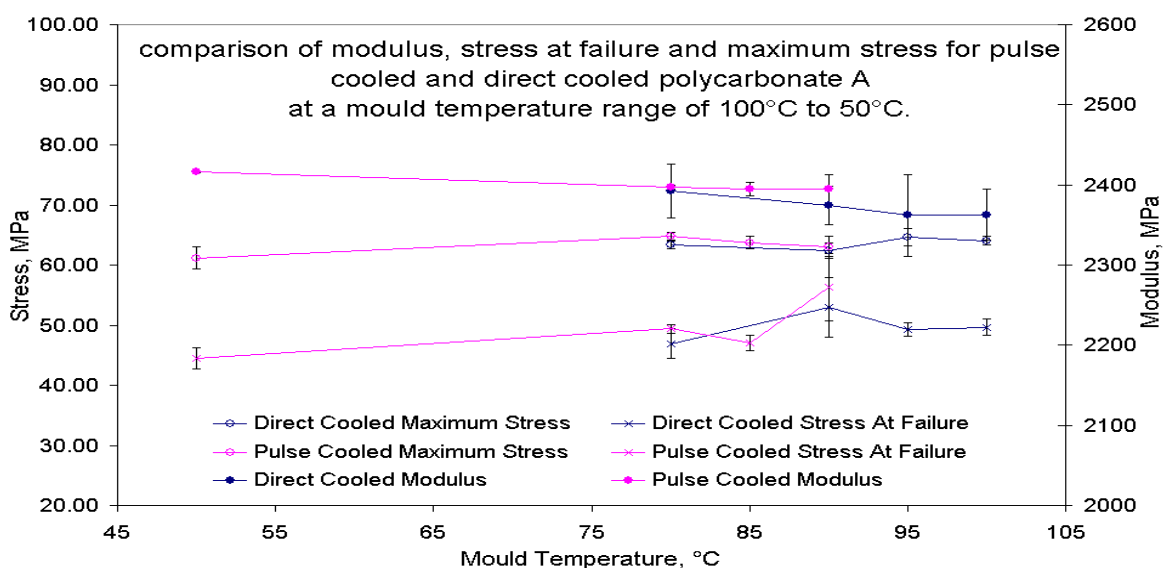


Figure 21 Comparison of modulus, stress at failure and maximum stress for pulse and direct cooled polycarbonate at a mould temperature range from 100°C to 50°C.

Direct cooled PC test bars were moulded with mould temperatures set at 100°C, 95°C, 90°C, 85°C and 80°C. Pulse cooled PC test bars were moulded with mould temperatures set at 90°C, 85°C, 80°C and 50°C. The mechanical properties of five left hand side direct cooled and five left hand side pulse cooled tensile test bars moulded at each temperature were compared. A comparison of pulse and direct cooled PC at 90°C and 80°C mould temperature produced no significant difference in the average stress to failure, modulus and maximum stress at each mould temperature for the two cooling methods (Figure 21), the error bars represent the standard deviation of the five test specimens. Therefore from comparing the modulus, maximum stress and stress at failure properties of direct and pulse cooled PC moulded over a range of mould temperatures, the mechanical behaviour for PC cooled by the two different methods was found to be similar.

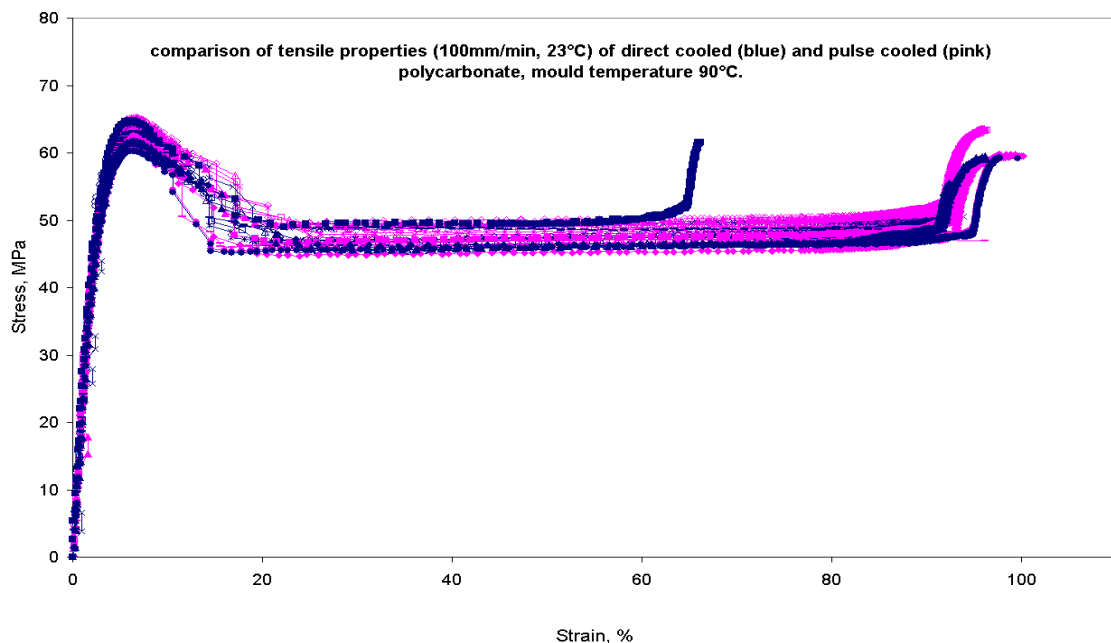


Figure 22 Comparison of the tensile properties of polycarbonate moulded at 90°C by direct and pulse cooled methods.

The stress/strain curves above are the results of tensile failure tests for pulse cooled and direct cooled polycarbonate injection moulded test pieces at a mould temperature of 90°C. The initial hump demonstrates yielding, followed by the associated load drop. There is then stable neck growth through cold drawing [9]. The increase in stress before eventual failure that is observed in some samples is due to the neck having travelled down through the test dumbbell to the start of the lower wider section of the test piece before failure. The cold drawing leads to failure at much greater strain than would be expected, so producing a tough material. These effects are seen in both pulse and direct cooled PC samples.

The 90°C mould temperature is below the glass transition temperature of PC. Therefore there is some regular arrangement of the polymer chains present within the sample. When the tensile test sample is subjected to a uniaxially strain, the polymer chains line up anisotropically along the axis of the test bar and extend, giving rise to cold drawing and the associated large strains at failure. Examination of the tensile test bars after stretching by using polarised light microscopy has confirmed the anisotropic behaviour, see Table 2.

Properties of Pulse and Direct Cooled Injection Moulded Polycarbonate Samples Examined Under A Microscope Under Plane Polarised Light And Between Crossed Polars					
Pulse Cooled			Direct Cooled		
sample	mould temperature, °C	behaviour between plane polarised light and crossed polars	sample	mould temperature, °C	behaviour between plane polarised light and crossed polars
NDB 200	90	before stretch (examined under plane polarised light): bright colours giving random patterns after stretch (examined between crossed polars): areas of brightness and extinction on rotation through 360°C	NDX 100	100	before stretch (examined under plane polarised light): bright colours giving random patterns after stretch (examined between crossed polars): areas of brightness and extinction on rotation through 360°C
NDC 200	85		NDY 100	95	
NDD 200	80		NDZ 100	90	
NDE 200	50		NDA 200	80	

Table 2 Behaviour of pulse and direct cooled polycarbonate test bars under polarised light.

Before stretching the moulded tensile specimens showed bright random colours under plane polarised light indicating that the molecular chains were arranged in a random manner within the material. After stretching, the tensile specimens showed areas of brightness and extinction on rotation through crossed polars. This indicated anisotropy within the material and that the polymer chains had lined up uniaxially under the tensile stress applied. This occurs for the polycarbonate samples cooled both by pulse cooling and direct cooling.

4.3 COMPARISON OF NYLON 6/CLAY NANO-COMPOSITE WITH VIRGIN NYLON 6 POLYMER

4.3.1 Differential Scanning Calorimetry

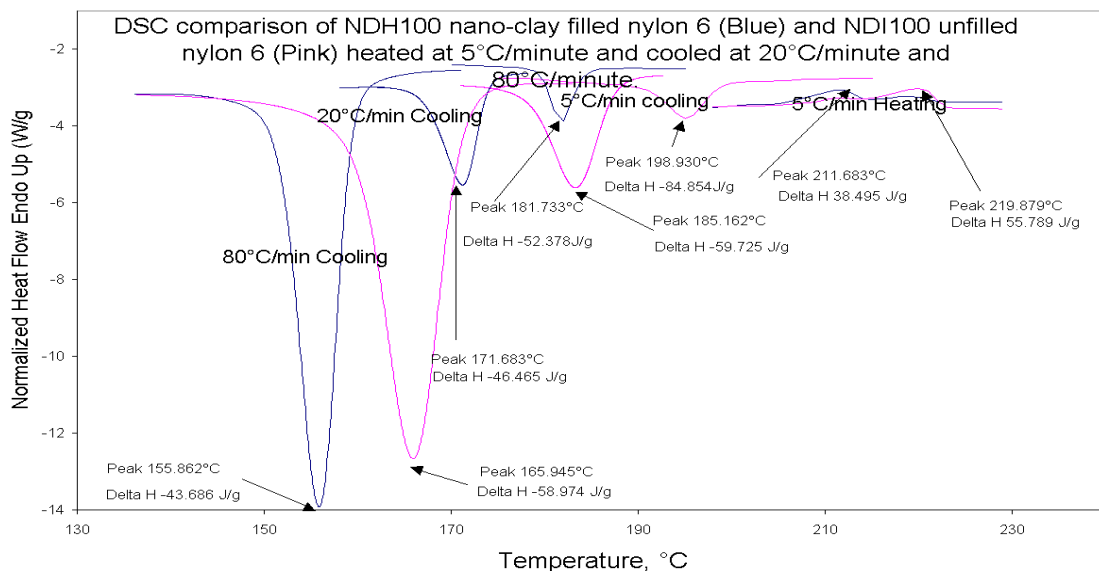


Figure 23 DSC comparison of nano – clay filled nylon 6 and unfilled nylon 6 at 5°C/min, 20°C/min and 80°C/min cooling rates and at a 5°C/min heating rate.

Figure 23 represents the DSC plots of both unfilled and nano-clay filled nylon 6 materials at 5°C/min, 20°C/min and 80°C/min cooling rates respectively. The peak temperatures and the

enthalpy data are shown in this case, which appeared to be noticeably different in the two materials. This shift in crystallisation temperature is in the opposite direction to that observed with traditional fillers, where nucleation of crystallisation is often seen. In the current study it is believed that a different nylon crystal structure (with a lower crystallisation temperature) is promoted by the presence of the clay.

4.3.2 Pressure/Volume/Temperature

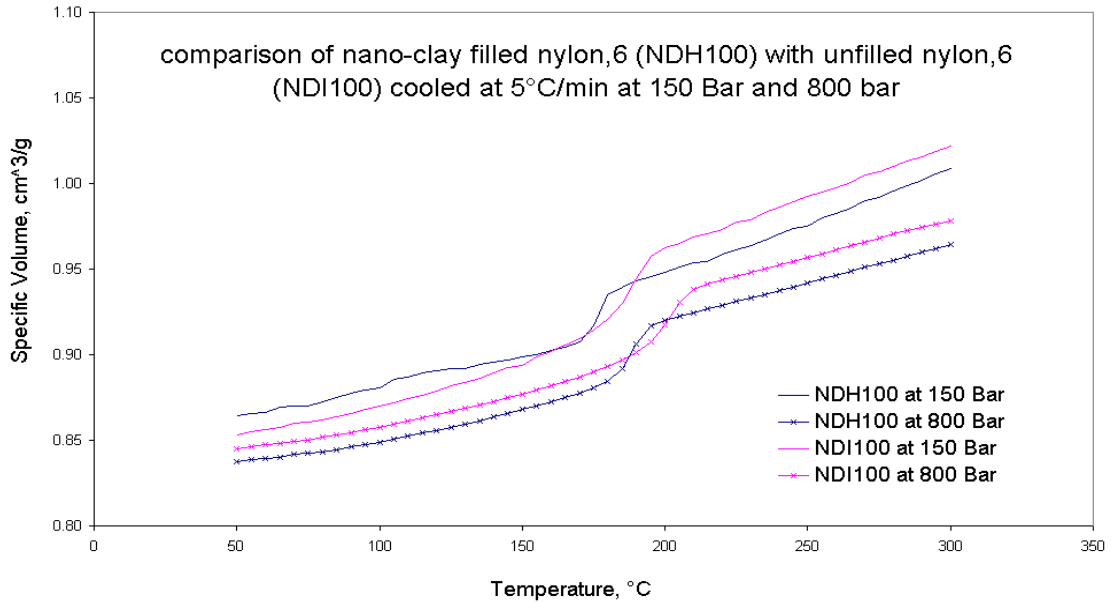


Figure 24 pvT comparison of nano-clay filled nylon 6 and unfilled nylon 6 cooled at 5°C/min under 150 bar and 800 bar pressures.

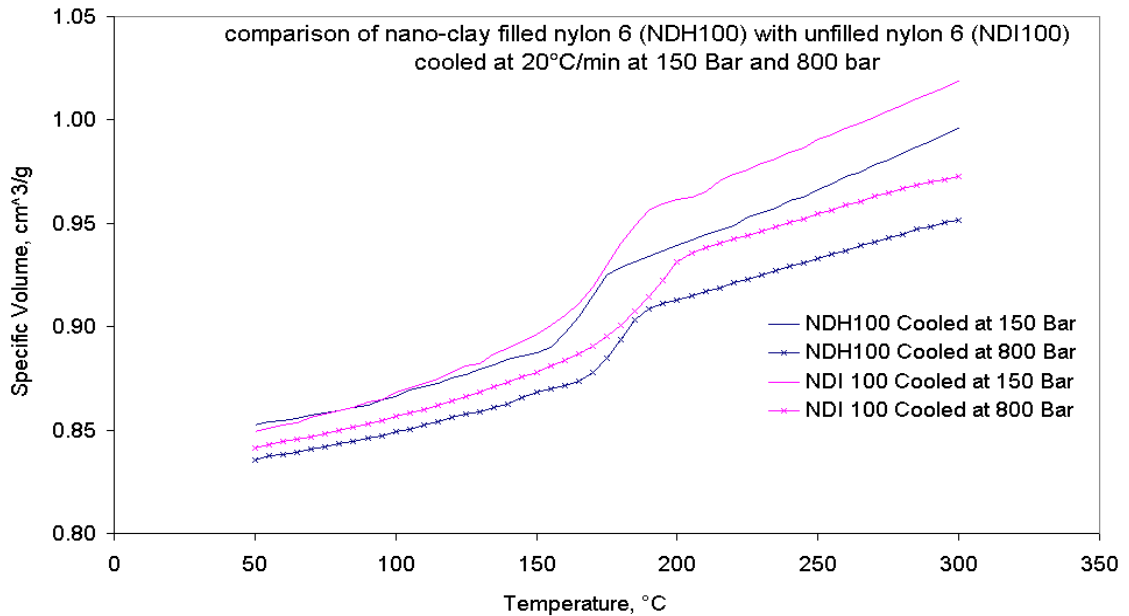


Figure 25 pvT comparison of nano-clay filled nylon 6 and unfilled nylon 6 cooled at 20°C/min under 150 bar and 800 bar pressures.

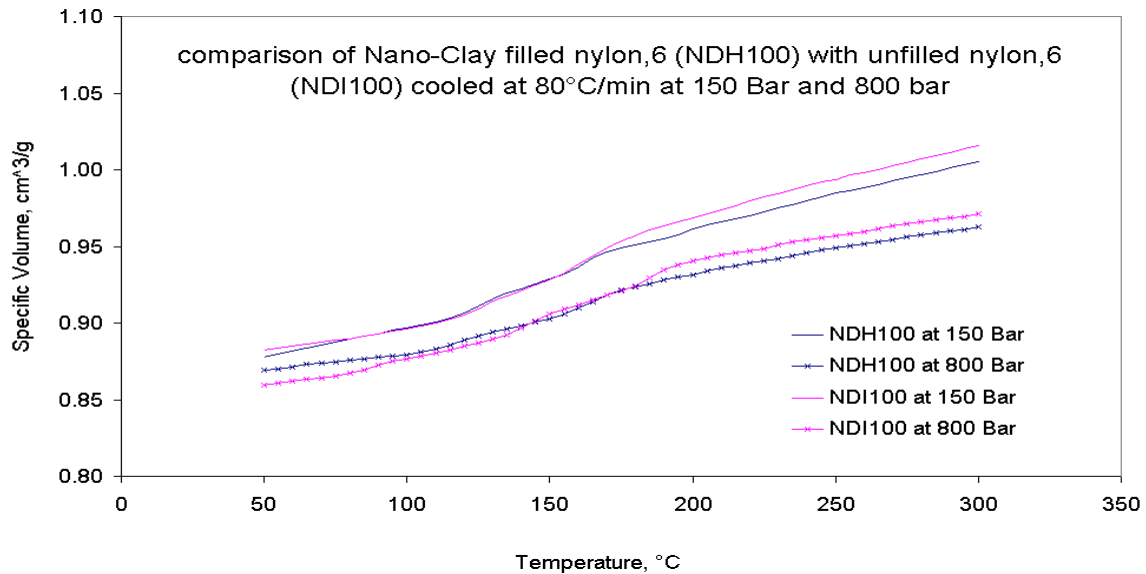


Figure 26 pvT comparison of nano-clay filled nylon 6 and unfilled nylon 6 cooled at 80°C/min under 150 bar and 800 bar pressure.

Iso-baric PVT tests were carried out at 150 and 800 bar on both unfilled and nano-clay filled samples at cooling rates of 5°C/min, 20°C/min and 80°C/min. The change in specific volume (ie shrinkage values) was measured as a function of temperature in the range 300°C to 50°C (Figures 24-26). In all cases, the nylon 6-clay nano-composite material showed an overall shrinkage of ~15%, compared to ~18% in the unfilled nylon 6. Melt compressibility was found to be of the same order in both cases i.e. ~5% at 300°C and ~2% at 50°C. The crystallisation temperatures were, as expected, found to be pressure dependant (varied by ~10°C), but in the case of nano-composites these crystallisation temperatures were reduced by about 10°C-15°C. These observations also agree reasonably well with the DSC results despite the difference of applied pressure between the experiments.

4.3.3 Thermal Conductivity

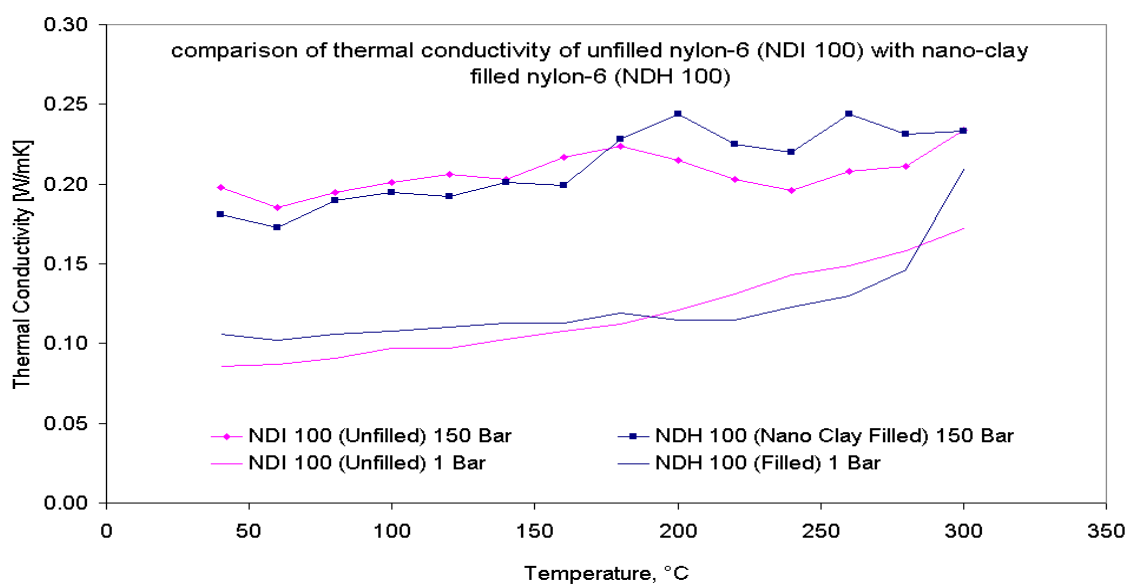


Figure 27 Thermal conductivity comparison of nano-clay filled nylon 6 and unfilled nylon 6 at 1 bar and 150 bar pressure.

No significant difference in thermal conductivity between the two materials was observed in the present study (Figure 27). However, both materials showed significant pressure dependency. Tests conducted at 150 bar gave thermal conductivity values about 80% to 100% higher than the values obtained at 1 bar in the temperature range 50°C to 300°C [10].

4.5 CRYSTALLISATION STUDY AT THREE DSC AND PVT COOLING RATES

4.4.1 High Density Polyethylene

4.4.1.a) DSC

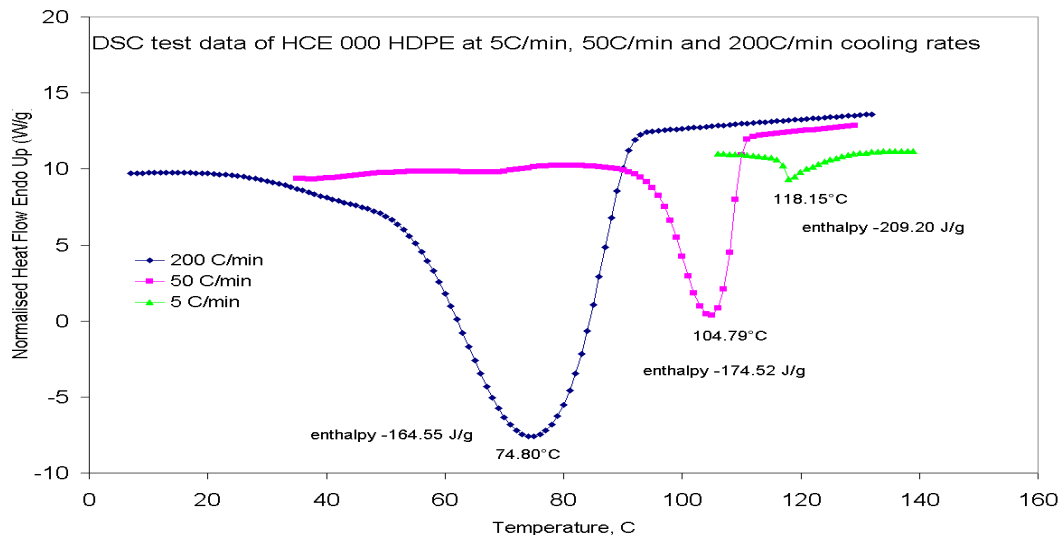


Figure 28 DSC test data for high density polyethylene cooled at 5°C/min, 50°C/min and 200°C/min cooling rates.

The DSC curves show how the crystallisation region of HDPE, which is sharp at 5°C/min, apparently becomes more diffuse as cooling rate increases. It can be observed that the final crystallinity of the polymer will be less at faster cooling rates. The temperature of crystallisation also decreases with cooling rate (Figure 28).

4.4.1.b) PVT

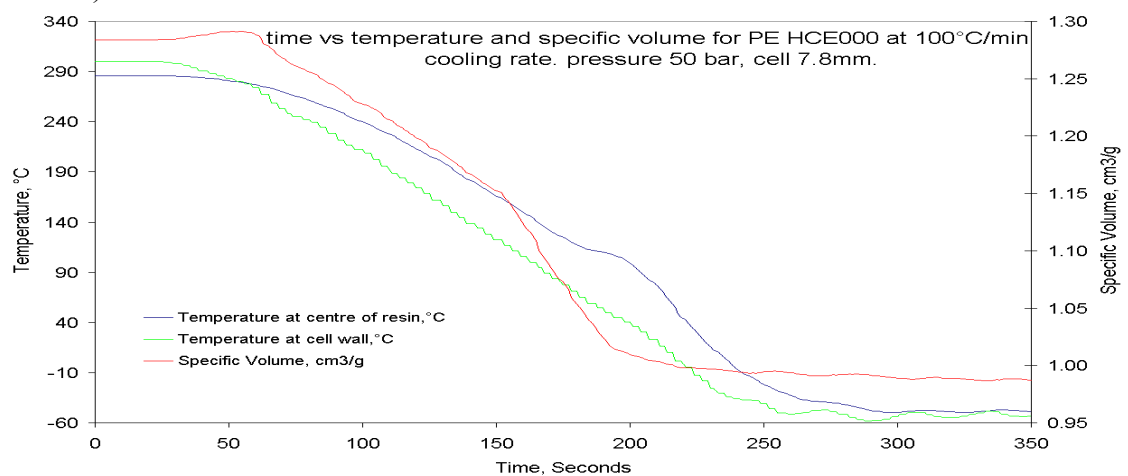


Figure 29 Time vs. temperature and specific volume for polyethylene at 100°C/min cooling rate and 50 bar pressure.

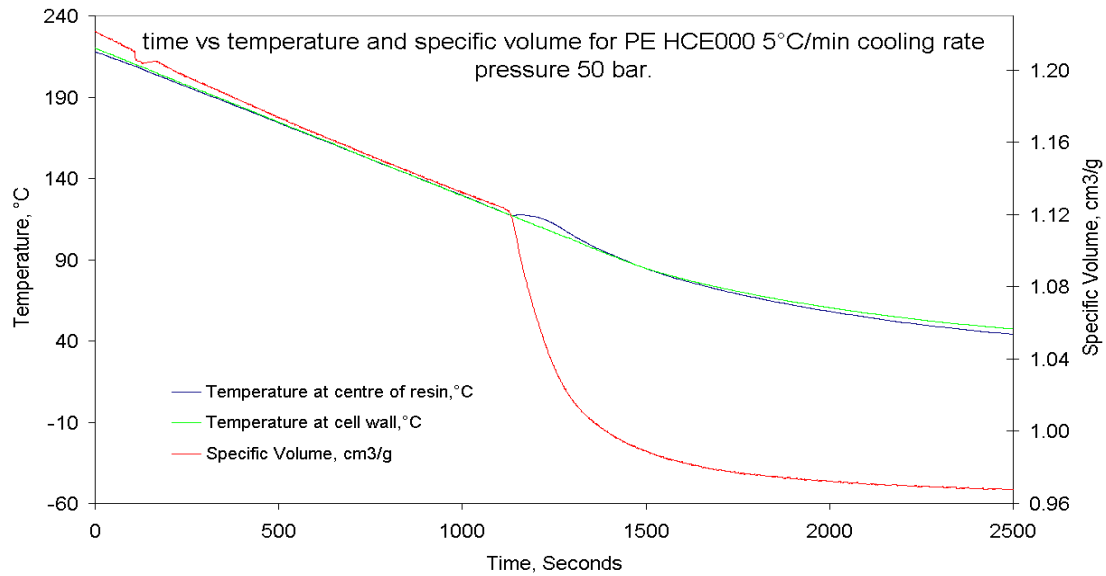


Figure 30 Time vs. temperature and specific volume for polyethylene at 5°C/min cooling rate and 50 bar pressure.

The HDPE PVT curves in Figure 29-30 show again how the crystallisation region, which is sharp at 5°C/min cooling, apparently becomes more diffuse as cooling rate increases to 100°C/min. The final crystallinity will be less at the faster cooling rate. Crystallisation considerably raises the overall shrinkage which is greater at the 5°C/min cooling rate than the 100°C/min cooling rate. From the time/temperature data, a small thermal lag between the temperature at the core of the sample and the temperature at the cell wall occurs at the faster 100°C/min cooling rate but is not apparent for the slower 5°C/min cooling rate.

4.4.2 Polypropylene

4.4.2.a) DSC

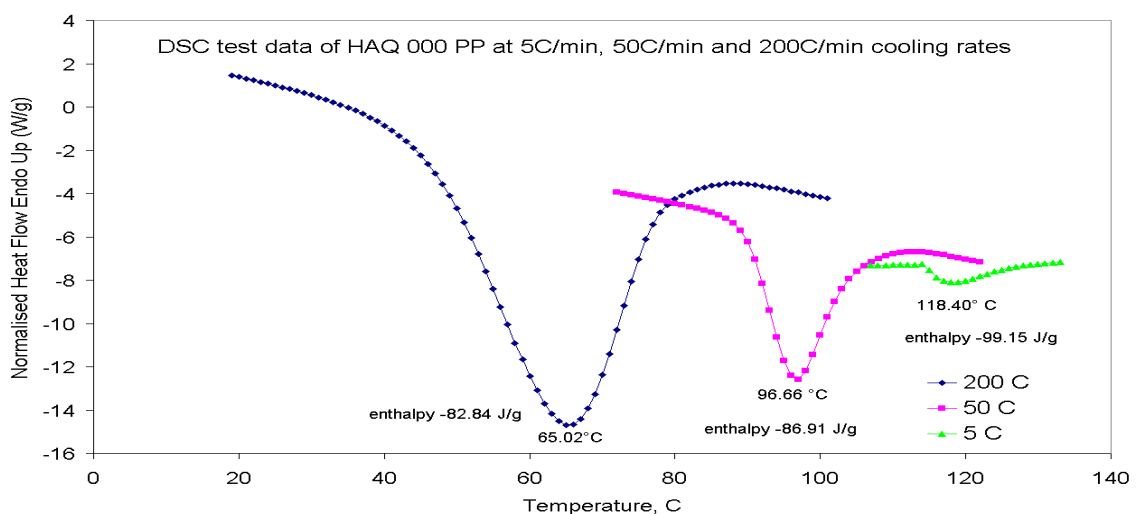


Figure 31 DSC test data for polypropylene cooled at 5°C/min, 50°C/min and 200°C/min cooling rates.

The DSC curves show how the crystallisation region of PP, becomes more diffuse as cooling rate increases. It can be observed that the final crystallinity of the polymer will be less at faster cooling rates. The temperature of crystallisation also decreases with cooling rate.

4.4.3 Poly(ethylene terephthalate)

4.4.3.a) DSC

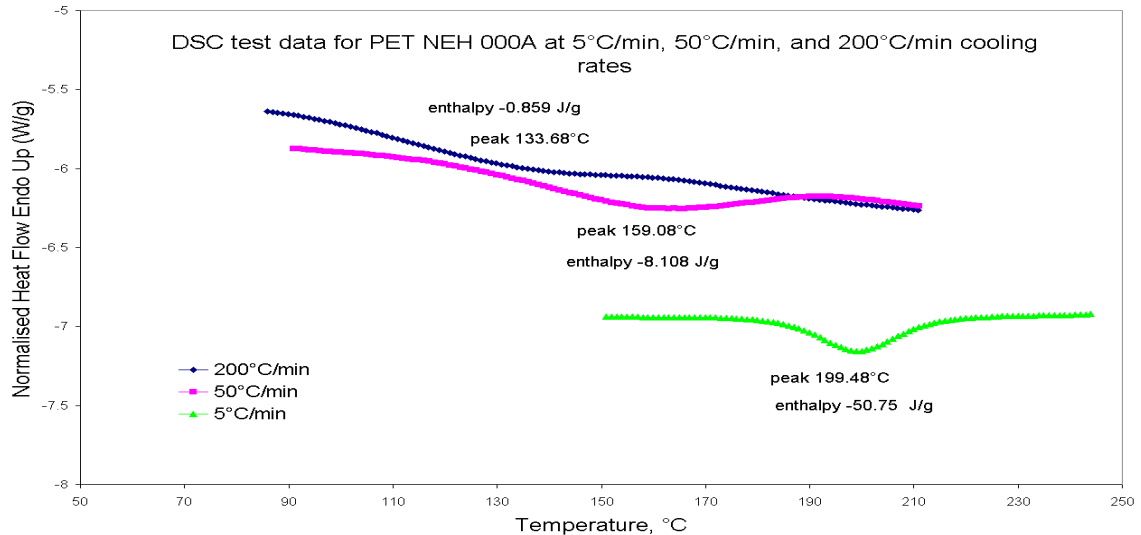


Figure 32 DSC test data for poly(ethylene terephthalate) cooled at 5°C/min, 50°C/min and 200°C/min cooling rates.

The DSC curves show that for PET, an amorphous polymer, at the slow cooling rate of 5°C/min, the final crystallinity of the sample will be low and will become even lower at the faster cooling rates of 50°C/min and 100°C/min. The temperature of crystallisation also decreases with cooling rate.

4.4.3.b) PVT

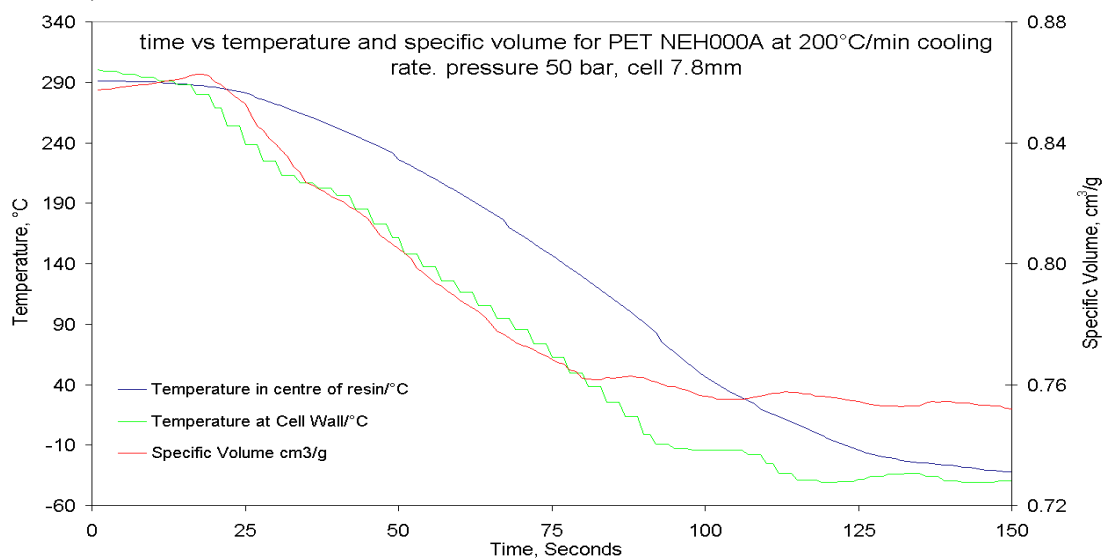


Figure 33 Time vs. temperature and specific volume for poly(ethylene terephthalate) at 200°C/min cooling rate and 50 bar pressure.

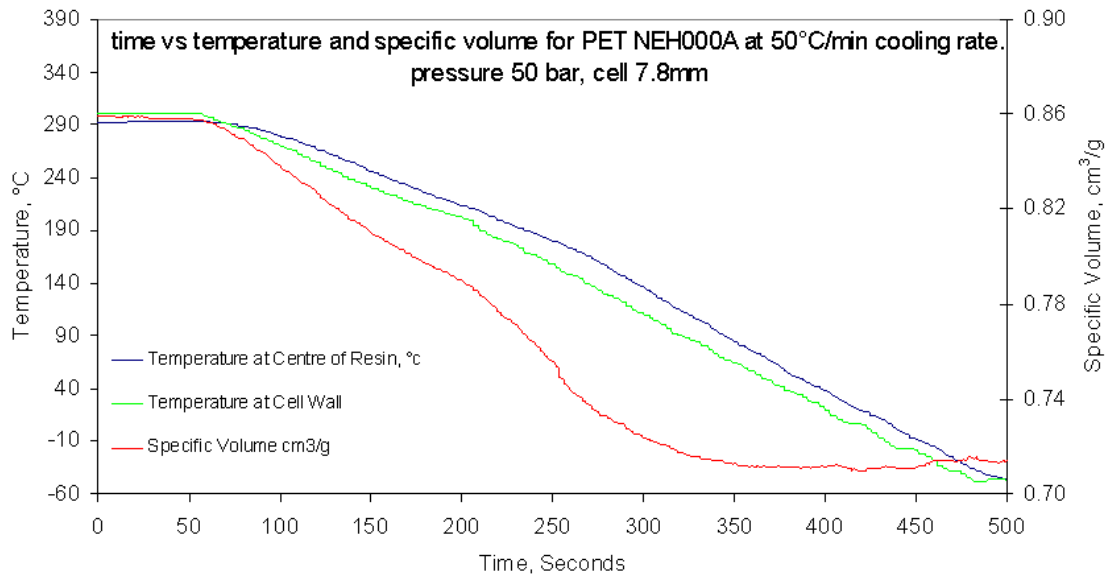


Figure 34 Time vs. temperature and specific volume for poly(ethylene terephthalate) at 50°C/min cooling rate and 50 bar pressure.

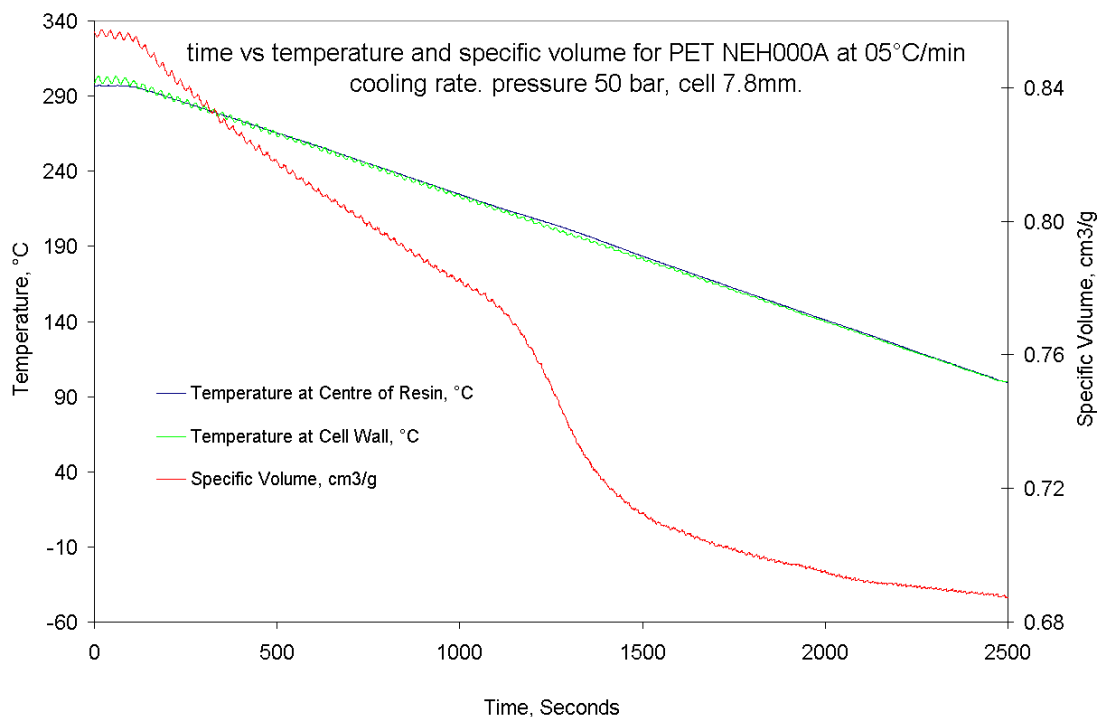


Figure 35 Time vs. temperature and specific volume for poly(ethylene terephthalate) at 5°C/min cooling rate and 50 bar pressure.

The PET PVT curves in Figure 33-35 show again how the crystallisation region, which is sharp at 5°C/min cooling, apparently becomes more diffuse as cooling rate increases to 100°C/min. The final crystallinity will be less at the faster cooling rate. The overall shrinkage for amorphous PET at slow cooling rates is relatively small as the material passes through the glass transition temperature to the glassy state. On ultrafast cooling the shrinkage is considerably less, so that the final glassy state has a higher specific volume and would exhibit different material properties as a result. From the time/temperature data, a small thermal lag between the temperature at the core of the sample and the temperature at the cell wall occurs at the faster 200°C/min and 50°C/min cooling rates but is not apparent for the slower 5°C/min cooling rate.

4.5 LATENT HEAT IN THE COOLING OF SEMI-CRYSTALLINE POLYMERS

A comparison between ABAQUS and an approximate analytical model for the cooling of a cylinder of material for constant and variable thermal property values was made.

Initially all material properties (thermal conductivity, specific heat and density) were held constant. From the analysis, predictions of centre-line temperature against time were obtained.

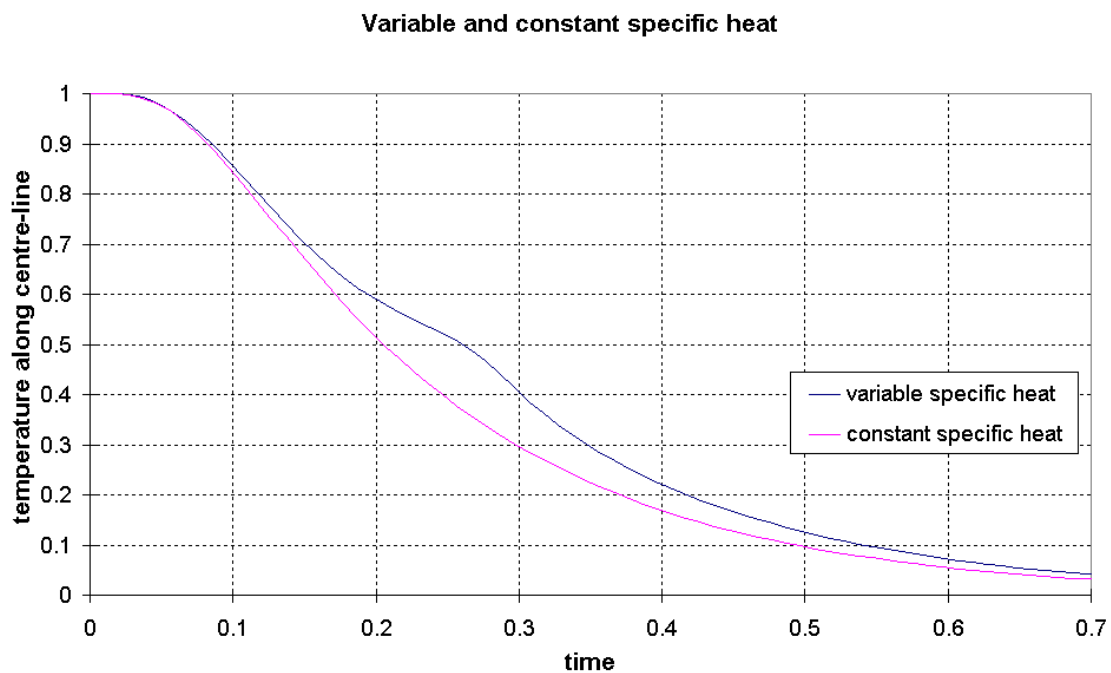


Figure 36 Predictions of temperature along centre-line variation with time for cooling of cylinder in variable specific heat and constant specific heat cases.

The results displayed in Figure 36 showed good correlation with the theoretical solution.

To incorporate variable properties, the equations from [11]

$$\beta(\theta) = 1 + A e^{-k(\theta-d)^2}$$

and

$$\beta(\theta) = pc/p_0c_0$$

were used to define the variation of specific heat with temperature. A , k and d are constants controlling the magnitude, spread and position of the peak in $\beta(\theta)$. p_0 and c_0 are the initial density and specific heat respectively. The data giving specific heat as a function of temperature were entered into the ABAQUS input file and the analysis rerun.

The predicted results were compared to those obtained from an approximate model. ABAQUS predictions were found to differ from those of the approximate analysis as shown in Figure 37.

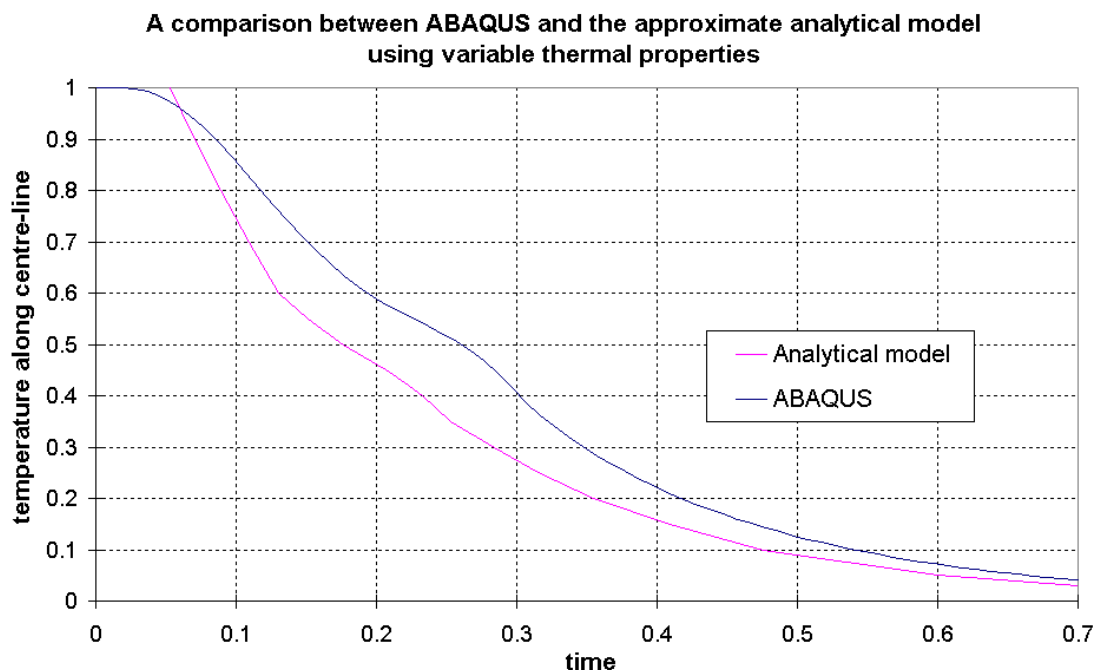


Figure 37 Comparison of predictions of temperature along centre-line variation with time for cooling of cylinder made using ABAQUS and the approximate analytical model using variable thermal properties.

ABAQUS predicted a slower decrease in centre-line temperature with time. Hence temperatures predicted by ABAQUS are consistently higher than the temperatures calculated by the approximate model, the greatest error being 40% at $t=0.25$. However the difference between the two values decreases with increasing time. A hump from the latent heat is visible in the plot, as in the approximate analysis but occurs at a slightly later time.

In predicting heat transfer in polymer processing, latent heat is often ignored. It was demonstrated that ignoring latent heat completely can lead to several errors in predicting heat transfer. Using DSC to measure latent heat, the greatest source of error, for polymers, was found to be baseline fitting. The effect of pressure was found to be ~10% from 1 to 1600

bar and was not considered a major problem as it agreed well with thermodynamic considerations. The effect of cooling rate can however, in some cases, lead to the latent heat disappearing altogether. For example in injection moulding of PET for bottles, cooling rates need to be fast enough to suppress crystallisation completely to ensure clarity. Techniques (DSC, PVT etc.) were developed to obtain thermal data in this metastable region.

In order to investigate the effect of data on specific heat and latent heat of crystallisation on cooling time predictions, the effect of three different sets of input data on how a simple 3.9mm radius cylinder of HDPE should cool from 255°C to room temperature was studied. Figure 38 shows the results.

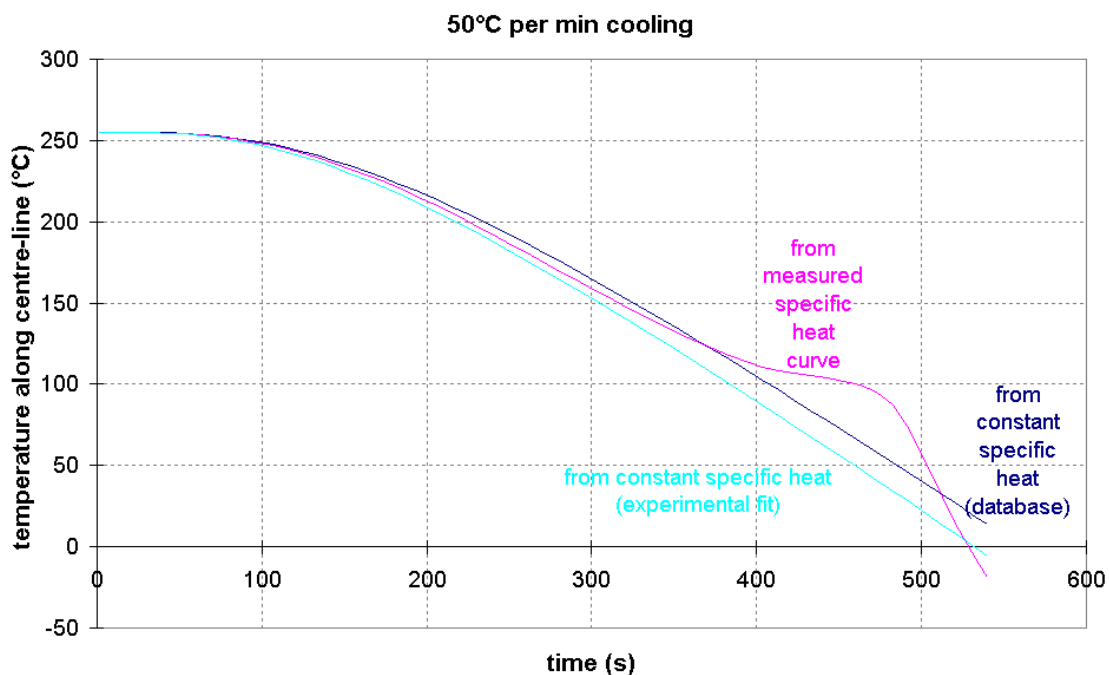


Figure 38 Effect of specific heat data from three different sources on the predicted cooling times of a 3.9mm radius high density polyethylene cylinder.

The result from using a single value of specific heat from a commercially available database is shown in dark blue. The specific heat was also measured at NPL using a DSC in cooling at 50°C/min. The pink curve in Figure 36 shows the predicted cooling curve using this data. A significant difference is seen particularly during the critical solidification phase around 100°C where delay of up to 58 seconds (or 13%) is predicted compared with the case which used a single value of specific heat from the database. Finally, the case of the best fit of a single value of specific heat to the experimental baseline data is shown in light blue (lowest curve). This shows an even bigger difference (up to 79 seconds).

This work demonstrates the significance of latent heat in the cooling of semi-crystalline polymer components and the difficulty of obtaining accurate predictions of cooling times without good data on the variation of specific heat with temperature.

5. DISCUSSION OF RESULTS

The polymer processing industry needs to operate at the shortest cycle times possible in order to achieve maximum production efficiency. The thermal conductivity and latent heat of crystallisation properties of a polymer can have a significant effect on the cooling times of a manufactured part and therefore cycle times.

The thermal conductivity results for HDPE, PP, PET and PS show the expected differences in thermal conductivity behaviour on cooling of amorphous and semi-crystalline polymers and also the differences in cooling behaviour between these materials within these two groups of polymers.

The thermal conductivity values gathered from these measurements can be used within a modelling package such as Moldflow to provide simulations to identify where any improvements may be possible.

Using the thermal conductivity instrument at NPL, measurements were obtained for polymers at temperatures and pressures similar to those applied during injection moulding. A comparison was made to compare the thermal conductivity of polypropylene with the thermal conductivity of polypropylene filled with Konduit at different proportions. Under constant pressure of 150 bar and across the temperature range from 250°C down to 170°C the thermal conductivity of the polymer increases with increasing proportion of Konduit. As a result of this increase in thermal conductivity, the cooling time of the filled material decreases with the increase in proportion of the conductive filler present.

The cooling time of polypropylene with Konduit decreases with increasing proportion of Konduit added for both direct and pulse cooling injection moulding methods. For the 25%, 50% and 75% levels of addition of Konduit the pulse cooled method leads to a larger decrease in cooling time than the direct cooling method. Undiluted Konduit reduced cycle time by 60% and no further reduction was observed with pulse cooling. Pulse cooling offered a 20% reduction in cycle time over direct cooling for polypropylene [5].

The addition of talc to polypropylene at levels of 10%, 20% and 30% by volume also lead to an increase in thermal conductivity of the polymer with an increase in proportion of filler. As in the case of the addition of Konduit to polypropylene, the addition of a commercially available filler improved the heat transfer properties of the polymer.

The addition of a nano-clay to nylon 6 did not significantly alter the thermal conductivity properties of the nylon 6. This might be accounted for by the low level of addition (2% by volume) of the clay filler to the nylon 6.

It was shown from the predicted cooling curve data for a PE cylinder based on specific heat data measured at NPL, that there is a 13% delay in cooling time during the critical solidification phase at around 100°C when compared to using a single value from a database. This difference is even larger (18%) when the data is compared to the best fit of a single value of specific heat to the experimental baseline data. Therefore latent heat is a significant

effect that should be taken into account when predicting the cooling time of components to be manufactured.

Reliable specific heat data in cooling mode is required to predict accurate cooling times of semi-crystalline polymers.

Poor knowledge of heat transfer properties can lead to higher than necessary scrap rates due to hot spots that cause degradation or excessive temperature gradients that lead to internal stresses and unacceptable warpage of products. An improved understanding of heat transfer will also lead to lower energy costs that will directly benefit the environment and minimise climate levy bills for the business.

These results have confirmed the importance of measurement methods for thermal conductivity, specific heat and latent heat in determining heat transfer properties. The effect of temperature and pressure have been shown to be significant as has the effect of conductive fillers. A key way to make use of this improved heat transfer data is through mathematical modelling.

For the moulded parts manufactured by pulse cooled and direct cooled injection moulding, the left hand side and right hand side tensile bars produced were found to be comparable.

Pulse cooled injection moulding of polypropylene and polypropylene filled with conductive aluminium filler showed a commercial advantage by reducing moulding cycle times while maintaining the mechanical performance (modulus, stress at failure) of the polymer at the same level as that of the direct cooled injection moulded material across a range of mould temperatures.

For polypropylene, after a study of sink marks, pulse cooling appears to produce tensile test bars with lower shrinkage than those produced by direct cooling.

For polycarbonate, the mechanical performance of mouldings (modulus, stress at failure, maximum stress) produced by pulse cooling was comparable to that of mouldings produced by the direct cooling injection moulding process. Toughening of the polycarbonate material occurs for both pulse cooled and direct cooled mouldings due to anisotropy within the material. Polycarbonate polymer chains line up uniaxially in the direction of the tensile stress.

Results from crystallinity studies in polymers provide important data for mathematical modelling. The output from the modelling can be used to predict the shrinkage occurring during a production process more accurately and so increase the cost effectiveness of the processing operation.

The two main measurement techniques used to provide data for crystallinity studies of polymers are DSC and PVT.

DSC results for nylon 6, nylon 6 nano-clay composite, HDPE, PP and PET all show that with increasing cooling rate of the polymer, crystallisation of the polymer occurs at increasingly lower temperatures and the degree of crystallinity (from enthalpy of crystallisation) decreases.

PVT studies for the same materials at two pressures confirm this. At faster PVT cooling rates the shrinkage (change in specific volume) is less than for slower cooling rates. This is because the degree of crystallinity decreases with increasing cooling rate. This is particularly important in the case of PET where at fast cooling rates the end material is amorphous, transparent and used in the production of drinks bottles. At slow cooling rates PET has a higher crystallinity and is opaque and white. The onset of crystallisation also occurs at lower temperatures as cooling rate is increased.

For all the materials studied there is good agreement between the onset of crystallisation temperature obtained by the DSC technique and the onset of crystallisation temperature obtained from PVT measurements, allowing for the difference in operating pressures.

An increase in pressure during PVT cooling measurements reduces the specific volume of the material and raises the temperature at which the material crystallises.

In the case of the DSC analysis of nylon 6 and nylon 6 nano-composite, it would be expected that the clay filler would act as a nucleating agent and cause the filled nylon 6 to crystallise at higher temperatures than the nylon 6. However, in the case observed, the addition of the clay to the nylon 6 suppresses crystallisation, lowering the temperature of crystallisation, so increasing the processing window of the nylon 6. This is thought to occur because of polymorphic behaviour exhibited by both the nylon 6 and the filled nylon 6. In nylon 6 cooling produces a higher ratio of the thermodynamically stable α crystalline form to the thermodynamically unstable γ crystalline form. The addition of a low concentration of clay filler increases the ratio of the γ crystalline form to the α crystalline form. This is due to the addition of clay altering the microstructure of the nylon 6 [12].

6. CONCLUSIONS

- Pulse cooling offers a 20% reduction in cycle time over direct cooling for polypropylene with no deterioration in physical properties
- Pulse cooling appears to reduce shrinkage in moulded polypropylene when compared with direct cooling
- Pulsed cooling offers a reduction in cycle time over direct cooling for Konduit filled polypropylene at levels of 25%, 50% and 75%
- Undiluted Konduit reduced cycle time by 60% with no further reduction for pulse cooling
- Pulse cooling of polycarbonate samples did not lead to a deterioration in mechanical properties of the moulded samples when compared to direct cooling

- Improvements in the modelling of heat transfer during plastics moulding processes can be made by measuring thermal conductivity and specific heat at conditions approaching those used during manufacture
- Improvements in the modelling of shrinkage during plastics moulding processes can be made by using PVT data obtained under near injection moulding pressures and cooling rates and fast cooled DSC data
- Improved modelling data will help to speed up production by better prediction of cycle times and make production more cost effective by reducing scrap rates due to degradation, warpage and shrinkage
- Adaptation of the PVT instrument to give ultrafast cooling data at injection moulding pressures gives realistic crystallinity data on crystallisation (or lack of crystallisation such as PET cooled at 200°C/min to give the clear, amorphous structure required for the manufacture of plastic bottles)
- Addition of 2% nano-clay to nylon 6 leads to an increased processability window for the filled polymer

7. REFERENCES

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9. APPENDIX

9.1 WORK PLAN

MATERIALS

Originally seven suggested materials were to be investigated:

- Polypropylene, unfilled (example of a crystalline material being used increasingly in engineering applications)
- Polycarbonate or acrylic resin (example of an amorphous engineering material)
- Carbon fibre reinforced material (special interest of BAe)
- Metal powder filled polymer to give high thermal conductivity
- Ceramic powder filled polymer to give low thermal conductivity
- One compound formulated to give maximum practical conductivity (probably containing fibres)
- One grade used by moulders in the consortium to produce critical components

These materials were amended in agreement with the customer to the more than seven materials detailed in the introduction to this report.

EXPERIMENTAL

1. PVT as a function of cooling rate

All materials will be characterised at three different cooling rates (5, 25 and 200°C/min) at six pressures (from 20Mpa to 250 Mpa). Total of 126 volumetric shrinkage curves.

2. DSC

The solidification of all materials will be measured by DSC at three cooling rates, matching the PVT cooling rates as closely as possible. This more traditional way of assessing crystallisation behaviour will be compared to the PVT method by extrapolating the PVT results to atmospheric pressure.

3. Thermal Conductivity

The thermal conductivity of the polymer melts will be measured using a line source probe method. This is an adaptation of the PVT apparatus which allows thermal conductivity to be measured at elevated pressure and temperature.

4. Latent Heat

The importance of latent heat to rapid processing will be investigated. Currently simulation models do not allow for latent heat effects when calculating temperature profiles. The size of the latent heat term in the heat transfer equations will be estimated and evidence for its effect in PVT curves sought.

5. Mechanical properties of unfilled pulse cooled and conventional samples at five different mould temperatures including a comparison of properties of left and right hand dumbbells.
6. Characterisation of sink marks in pulse cooled and conventional unfilled samples.
7. Measurement of mechanical properties of polycarbonate and Konduit filled pulse cooled and conventional samples. (Polycarbonate substituted talc filled samples with agreement of customer).
8. Measurement of thermal conductivity and DSC of unfilled and nano-particle filled nylon.
9. In addition to the practical work there has also been a requirement to prepare papers, presentations, input to the final report, input to the mould design and attend meetings.

Items 5 to 8 replaced 3, 4, 5, 7 and A of original work plan with customer agreement.

In addition to specific analysis related to each work package it was necessary to analyse results as a whole and to write a report and at least one scientific paper on the results. Considerable interaction with partners within the consortium was required to ensure the success of the whole project. The work packages and interaction with other partners was project managed in accordance with the NPL quality system (accredited by Lloyds to ISO 9001).

OTHER PROJECT DETAILS

End date: 30 September 2003
Starting date: 1 February 2000