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Investigation of Thermal Stability of Non-Newtonian Melt Flows

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Abstract – Polymers are one of the major classes of raw materials available for many aspects of production. Extrusion is a fundamental method for processing polymeric materials to form a wide range of products. Being poor thermal conductors these materials are difficult to heat and cool and also exhibit high viscosity in their molten state. Hence, heat transfer and rheology are major aspects of polymer processing applications. Also, polymer melts usually exhibit a non-Newtonian behaviour and hence are quite complicated to model. This work aims to observe the thermal behaviour/quality of a number of industrially common polymeric materials under a wide range of processing conditions using a capillary die with a specifically designed thermocouple mesh sensor. The melt temperature is considered as the main parameter in determining the thermal quality of the melt. Materials were processed under different screw speeds and set temperature conditions using three different screw geometries. The results showed that the uniformity of temperature across the melt flow varied considerably with increase in screw rotational speed whilst it was also shown to be dependent upon process settings, screw geometry and material properties. Moreover, it appears that the effects of the material, machine and process settings on the quantity and quality of the process output are heavily coupled with each other and this may cause the process to be difficult to predict and variable in nature. In terms of the flow behaviour, regardless of the processing conditions used, the highest melt temperature was observed always in the middle of the melt flow. At lower processing speeds, the lowest melt temperature was close to the die wall where this position shifted a few millimetres away from the die at higher speeds by forming low temperature shoulder regions (making the melt flow highly thermally inhomogeneous). Moreover, a discussion was made on the possible effects of materials' properties on viscous heat generation during processing. Additionally, the importance of the use of the appropriate thermal monitoring techniques can also be highlighted in determining the actual melt thermal quality during polymer processing applications.

***Keywords*:** Melt temperature, Polymer processing, Process monitoring, Non-Newtonian flow, Fully-developed flow, Thermal stability, Shear rate, Shear heat, Power-law index, Brinkman number.

1. Introduction

Currently, many conventional raw materials (e.g., metal, glass, wood) have been replaced with advanced polymer materials, particularly in the packaging and automotive sectors, due to their efficiency in energy saving. Consequently, PlasticsEurope [1] emphasised the importance of polymeric materials by quoting the headline: *'Plastics – the materials for the 21st century'*. Extrusion is the fundamental technique for processing polymeric materials; for example, virtually all thermoplastics are compounds which are produced by extrusion mixing and the majority of these materials are also formed into their final product shape by extrusion. However, the flow behaviour within polymer processing equipment is not yet well understood and hence these processes are highly unpredictable in nature. Moreover, it is often stated that the majority of polymer processing units are kinds of 'black boxes', as it is very difficult to obtain real-time information on the processing conditions and polymer flow behaviour inside these units. Given these issues, these processes usually operate under conservative rates with considerable wastage of raw materials, labour, time, etc. while also resulting in poor thermal and energy efficiencies. Despite considerable research and development over the last few decades, process monitoring and control in the majority of polymer processes still remain problematic and process operators have to deal with issues such as the selection of process settings and product quality control primarily by trial and error. Usually, the thermal homogeneity of the process melt output presents a major challenge for high quality extruded products. Although the process energy efficiency increases with the processing speed, it is a problem to operate these processes at high speeds as the thermal efficiency deteriorates with the processing speed. In this case, one of the major issues is the difficulty of determining the actual spatial and temporal thermal dynamics/information across the process output melt flow. Moreover, the modelling of the melt flow behaviour is challenging due to their high viscosity and non-Newtonian nature.

1.1. Non-Newtonian behaviour of polymer melts

In general, the viscosity of non-Newtonian fluids depends on shear rate. It has been proposed that polymer melts behave as Newtonian fluids at shear rates below about 10^{-1} s and as non-Newtonian fluids at higher shear rates [2]. As was stated by Vlachopoulos [3], the shear rates may reach up to 200 s^{-1} in the screw channel near the barrel wall, and can be significantly higher between the flight tips and the barrel during single-screw extrusion. Moreover, at the lip of the die the shear rate can be as high as 1000 s^{-1}. Hence, it is quite clear that melt flows exhibit a non-Newtonian flow behaviour in polymer extrusion and also in the majority of other polymer processing techniques. Furthermore, polymer melts are usually shear-thinning fluids which is also known as pseudo-plastic behaviour therefore the higher the shear rate, the easier it is to force polymers to flow through extrusion dies. However, high shear rates also promote melt flow instabilities such as sharkskin (or surface mattness) and melt fracture.

In polymer processing, it should be ensured to achieve the complete filling of the mould before a significant chemical reaction begins and also the achieving of a better mixing of the melt is important to ensure uniform mechanical properties from the products. Based on the basic fluid flow principles, a laminar flow is desired for smooth mould filling while a turbulent flow would be good in enhanced mixing performance. However, turbulent flows may create problems such as air bubbles leading to the porosity of the structure hindering mechanical properties. Hence, a kind transitional flow would be desirable with high viscous polymer melts, however the nature of flow should be determined based on the factors such as the materials' viscosity, product geometry, size and complexity to achieve better mixing and mould filling simultaneously. The achievement of thermally homogenous melt flow is also one of the crucial requirements in polymer processing while achieving a good mixing and a complete mould filling. Hence, the study of the thermal quality of polymer processing is an investigation of the thermal properties of fluids which exhibit non-Newtonian and transitional flow behaviours.

1.2. Melt flow thermal homogeneity

The main function of an extruder is to deliver a homogeneous polymer melt at a specified uniform temperature and pressure. Process output is required to be homogenous in composition, colour and temperature. For this purpose, extruders are generally equipped with an efficient drive and feed system, a screw designed to melt and convey the polymer and devices such as temperature and pressure sensors to monitor the system for troubleshooting and control. Additionally, several other auxiliary devices may be used (e.g., mixers, gear pumps and controlled feeding devices) based on the nature of the process/product. Although, melt quality (defined as a thermally homogeneous melt at a constant throughput) is a key variable in polymer extrusion only a few thermal monitoring techniques are able to determine thermal stability and homogeneity across the melt flow cross-section in real-time (see Table 1). Therefore, extrusion processors may have limited understanding of the actual temporal and spatial thermal behaviour/fluctuations across the melt flow cross-section and may be unaware of the effects of radial thermal fluctuations on their product quality and processing problems.

1.3. Melt temperature measurements

At present, wall-mounted thermocouples are the most commonly used melt temperature measurement technique in polymer processing [4, 5]. Additionally, infrared sensors and resistance temperature detectors are also used by some manufactures. A summary of current industrial melt temperature measurement techniques is presented in Table. 1.

Table 1: Summary of the polymer extrusion thermal measurement techniques available in industry.

NO.	Technique	Measurement		Non- Invasive	Accuracy	Response time
		Point	Profile			
1	Wall mounted thermocouples	√		Yes/No	Low	~ 1 s
2	IR	√		Yes	Medium	~ 10 ms
3	Ultrasonic probe	√		Yes	Medium	~ 1 ms
4	Auto-traversing probe		√	No	Low	~ 1 s
5	Thermocouple mesh		√	No	High	~ 0.1 s (6 ms)
6	Fluorescence technique		√	Yes	Medium	-

More details on the thermal measurement techniques available in industry and research were discussed by the authors previously together with an experimental evaluation of some of them [5-7].

The aim of this work was to explore melt temperature homogeneity across a range of extrusion conditions to provide an improved understanding of the individual and combined effects of various processing conditions (such as screw geometry, material, and process settings (screw speed, barrel/die set temperatures)) on process thermal homogeneity and flow behaviour. Six commonly used commercial grade thermoplastics and three different screw geometries were used for the experiments at five discrete screw speeds and three temperature settings (Low, Medium and High). The study was focused on single screw extrusion and the experiments were performed to replicate industrial processing conditions by covering the full operating range of the extruder (i.e. 0-100 rpm).

2. Experimental procedure and materials

All measurements were carried out at the University of Bradford on a highly-instrumented Davis Standard BC-60 63.5 mm diameter (D) single-screw extruder. Three different screws were used for the tests: a gradual compression (GC) screw, a rapid compression (RC) screw, and a barrier-flighted (BF) screw with a Maddock mixer. The geometrical details of the screws (i.e. compression ratio (CR) and length (L) of each section and channel heights (H) as functions of screw diameter) are given in Table 2. The extruder was fitted with a 38 mm diameter adaptor prior to a short cylindrical die with a 12 mm bore. The extruder barrel has four separate temperature zones with another three separate temperature zones at the clamp ring, adapter and die. Each of these temperature zones is equipped with a separate temperature controller which allows individual control of the set temperature. The arrangement of the apparatus (i.e., extruder barrel, adapter and die) is shown in Fig. 1-(a). In all experiments, melt temperature profiles at the adapter (i.e., prior to entering the 12 mm die) were measured using a thermocouple mesh (TCM) placed in-between the adapter and die as shown in Fig. 1-(b).

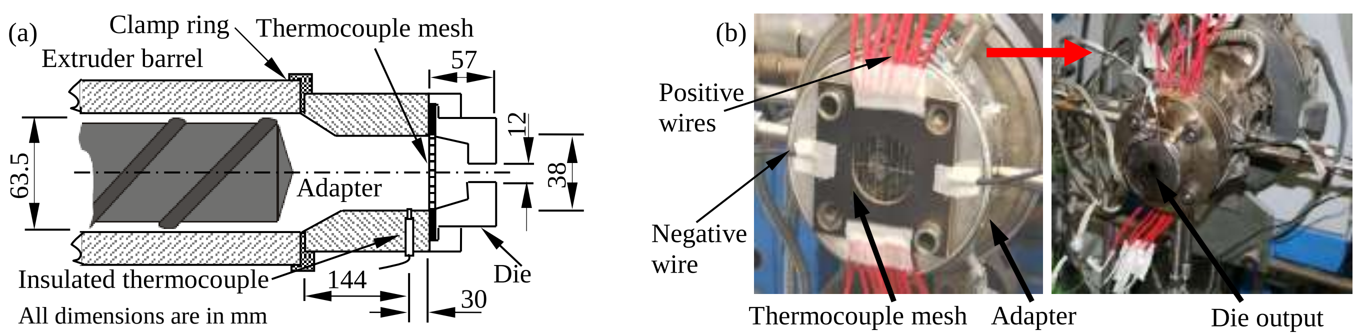

Fig. 1: (a) - Arrangement of the apparatus with its dimensions, (b) - Images of the thermocouple mesh placement.

Table 2: Geometrical details of the screws used for experiments.

Screw	CR	Feed		Melting	Metering	
		L	H (mm)	L	L	H (mm)
GC	3:1	4D	10.53	10D	10D	3.46
RC	3:1	12D	10.53	2D	10D	3.50
BF	2.5:1	5D	12.19	13D	6D	4.90

As previously reported by Brown el al. [8] and Kelly et al. [9], die melt temperature measurements are symmetrical across the centreline of the thermocouple mesh when averaged over sufficient time. Therefore, the thermocouple junctions (i.e., between a number of positive wires and the negative wire) were placed asymmetrically across the melt flow along the diameter of the mesh as shown in Fig. 1.(b), and then the complete die melt flow temperature profile can be obtained by mirroring these reading over the centreline. Moreover, an insulated wall mounted thermocouple (0.5 mm in diameter and flush mounted to the wall) was used to measure the temperature of the melt close to the die wall and this measurement was used as the melt temperatures of the ±19 mm radial positions for generating the melt temperature profiles across the melt flow. Four different thermocouple meshes were used and details are given in Table 3.

Table 3: Details of the thermocouple mesh used for experiments.

TCM	Distance from die centre to each mesh junction (mm)	No. of junctions	Materials processed
1	0, –2.5, +5.0, +8.0, +11.0, –14.0, +16.5	7	LDPE-G1, LLDPE, PS
2	0, –2.5, +4.3, +8.5, +11.0, –15.0, +16.8	7	LDPE-G2
3	0, –3.0, +4.3, +9.0, +11.0, –15.0, +17.0	7	HDPE
4	0, –2.5, +3.5, +9.0, +11.3, –14.5, +16.7	7	PP

A data acquisition programme developed in LabVIEW was used to communicate between the experimental instruments and a PC, and all signals were acquired at 10 Hz sampling speed.

2.1. Materials and experimental conditions

Six unfilled thermoplastics were used for the experiments: a polystyrene (PS); two different grades of low density polyethylene (LDPE-G1 and LDPE-G2); a linear low density polyethylene (LLDPE); a high density polyethylene (HDPE); and a polypropylene (PP); and the details of these materials' properties are given in Table 4.

Table 4: Details of the materials used for experiments.

Property	Material					
	LDPE-G1	LDPE-G2	LLDPE	HDPE	PP	PS
Trade name	LUPOLEN 2420H	DOW-LD150R	FLEXIRENE CL10	INEOS HM5411	INEOS 100-GA03	STYROLUTION PS 124N
Density (g/cm^3)	0.924	0.921	0.918	0.952	0.952	1.040
Melt flow index (g/10 min)	1.9 (190 °C, 2.16 kg)	0.25 (190 °C, 2.16 kg)	2.6 (190 °C, 2.16 kg)	0.12 (190 °C, 2.16 kg)	3.00 (230 °C, 2.16 kg)	-
MVR @ (200 °C, 5 kg)	-	-	-	-	-	12 cm^3/10 min
Melt temperature (°C)	111	111	121	134	163	-
Vicat softening temperature (°C)	94	-	97	-	-	87
Morphology (all in pellets form)	Semi-crystalline	Semi-crystalline	Semi-crystalline	Semi-crystalline	Semi-crystalline	Amorphous

Table 5: Extruder barrel temperature settings.

Test	Temperature settings (°C)						
	Barrel zones				Clamp ring	Adapter	Die
	1	2	3	4			
A	130	155	170	180	180	180	180
B	140	170	185	200	200	200	200
C	150	185	200	220	220	220	220

Three different extruder temperature settings were selected as described in Table 5 and denoted as A (low temperature), B (medium temperature) and C (high temperature). These settings were selected by considering the material properties and the screw geometry to achieve normal process operating conditions throughout the experiments while covering a wide operating window of the extruder. The overall time of the each test was around 45 minutes and the extruder was allowed to stabilize after each step change in screw speed. The average melt temperatures over the last minute at each speed were used to plot the melt temperature profiles presented in section 3.

3. Results and Discussion

The effect of screw geometry, materials and process seetings were investigated on the temeperature profile of the melt flows and discussed in this section. Heat generated in the high shear rate regions near the wall is conducted towards the centre of the tube and progressively the temperature profile changes towards an equilibrium profile, which has a maximum at the centre of the channel due to significant conduction to the walls. The temperature profile is generated is thus a function of heating due to viscous dissipation and thermal conduction to/from the heated metal wall. For the simple case of unidirectional shear the viscous heat generation per unit volume, p (W m^{-3}) for a power-law fluid is given by Eq. (1):

$$p = K\dot{\gamma}^{n+1} \tag{1}$$

where $\dot{\gamma}$ is the shear rate, K is the consistency index (a measure of the polymer's thermoplasticity) and n is the power-law coefficient (a measure of the polymer's pseudoplasticity). The local rate of heat generation will therefore vary with the local shear rate which, due to the low thermal diffusivities of polymer melts, can result in distinct thermal gradients as seen in the experimental temperature profiles. In this study, the flatness of the temperature profile was considered as a measure of the melt temperature homogeneity/quality where the flatter the profile the higher the thermal quality of the melt.

3.1. Effects of screw geometry

As shown in both Figs. 2 and 3, a reduction in profile flatness and the development of a peak in the centre of the melt flow were observed with increasing screw speed. Of the three screw geometries, the BF screw showed the flattest melt temperature profiles (indicating better thermal homogeneity), and also showed the smallest variation in melt temperature with increasing screw speed. These data agrees with previous studies by Kelly et al. [9, 10] and Abeykoon et al. [6, 11-15]. The separation of melt and solid into separate channels in the BF screw and the spiral Maddock mixer at the end of the screw are thought to be responsible for the improved melt flow homogeneity [16]. In addition, melt temperature profiles across the melt flow cross-section after the BF screw do not exhibit shoulder regions at 70 and 90 rpm speeds unlike the profiles observed with the single-flighted GC and RC screws which give reduced melt temperature homogeneity. Therefore, it is clear that screw geometry has a significant effect on the magnitude of spatial variations in the melt temperature in polymer extrusion.

Screw geometry was found to affect both the magnitude and distribution of the melt temperature across the flow. Of the three screws, at set temperature condition A, the BF screw exhibited the highest maximum melt temperature while the lowest melt temperature occurred with the RC screw, both at 90 rpm. However, at the higher set temperature conditions of B and C, the highest and the lowest melt temperatures are reported at 10 and 90 rpm respectively regardless of the screw geometry used. Heat generation due to viscous dissipation (shear heating) may be expected to increase with shear rate and therefore screw speed, resulting in higher melt temperatures. However, it is quite interesting to see that the maximum melt temperature attained gradually reduced from 10-90 rpm at the higher set temperature conditions of B and C for all screws for this particular LDPE material (G1). Polymers with a low power-law coefficient such as the LDPE- G1 ($n = 0.39$) will generate less heat by viscous dissipation in the melt with increasing shear rate as screw speed is increased from 10 to 90 rpm. If viscous dissipation becomes limited compared to conduction, as is increasingly the case here as the screw speed is increased, the temperature profile near the wall is increasingly dictated by the temperature of the wall. In addition, given the slow rate of thermal conduction, as the mass throughput increases with screw speed the influence of the lower temperature settings of the upstream heating zones begins to be observed and the average melt temperature drops. According to Fig. 2, the highest magnitude of variation in melt temperature (i.e., the difference between the highest and lowest melt temperatures) was observed at the set temperature condition C for all the screws, giving a value of around 50 ˚C with the GC screw at 90 rpm, 47.5 ˚C at 90 rpm for the GC screw and 23 ˚C at 10 rpm for the BF screw. As was reported in the literature [17], the temperature of the screw has a considerable impact on the melting process. Although the same material and process settings were used in this experiment, the surface temperature of the screw may be variable since the mechanical heat generation is a function of screw geometry. However, screw temperature cannot be measured in this extruder arrangement used for experiments. The rate of mass throughput (i.e., melting capacity) and the level of material mixing also depend on the screw geometry [18, 19] and will also impact upon the heat transfer and contribute to the measured melt temperature profiles.

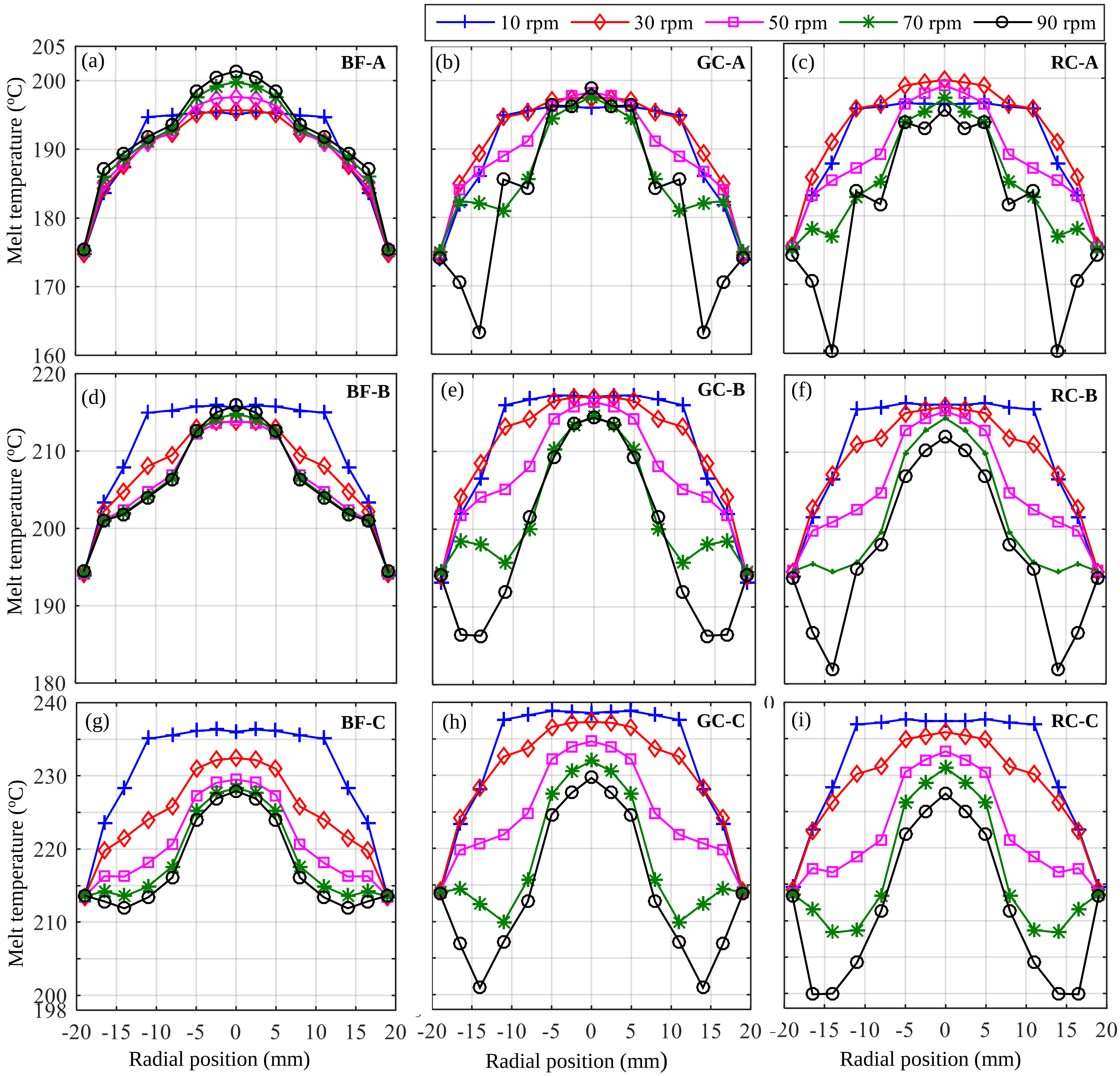

Fig. 2: Melt temperature profiles across the 38 mm diameter die for LDPE-G1with different screw geometries (sub-figures in each row are at the same scale).

3.2. Effect of materials

The properties of feedstock materials (e.g., viscosity, surface proprties, melting temperature) also can have a significant influnce on the melt thermal stability and is evident from Fig. 3. All six materials shown in Fig. 3 were processed at the set temperature condition B and the sub-figures in the same row relate to the same screw geometry.

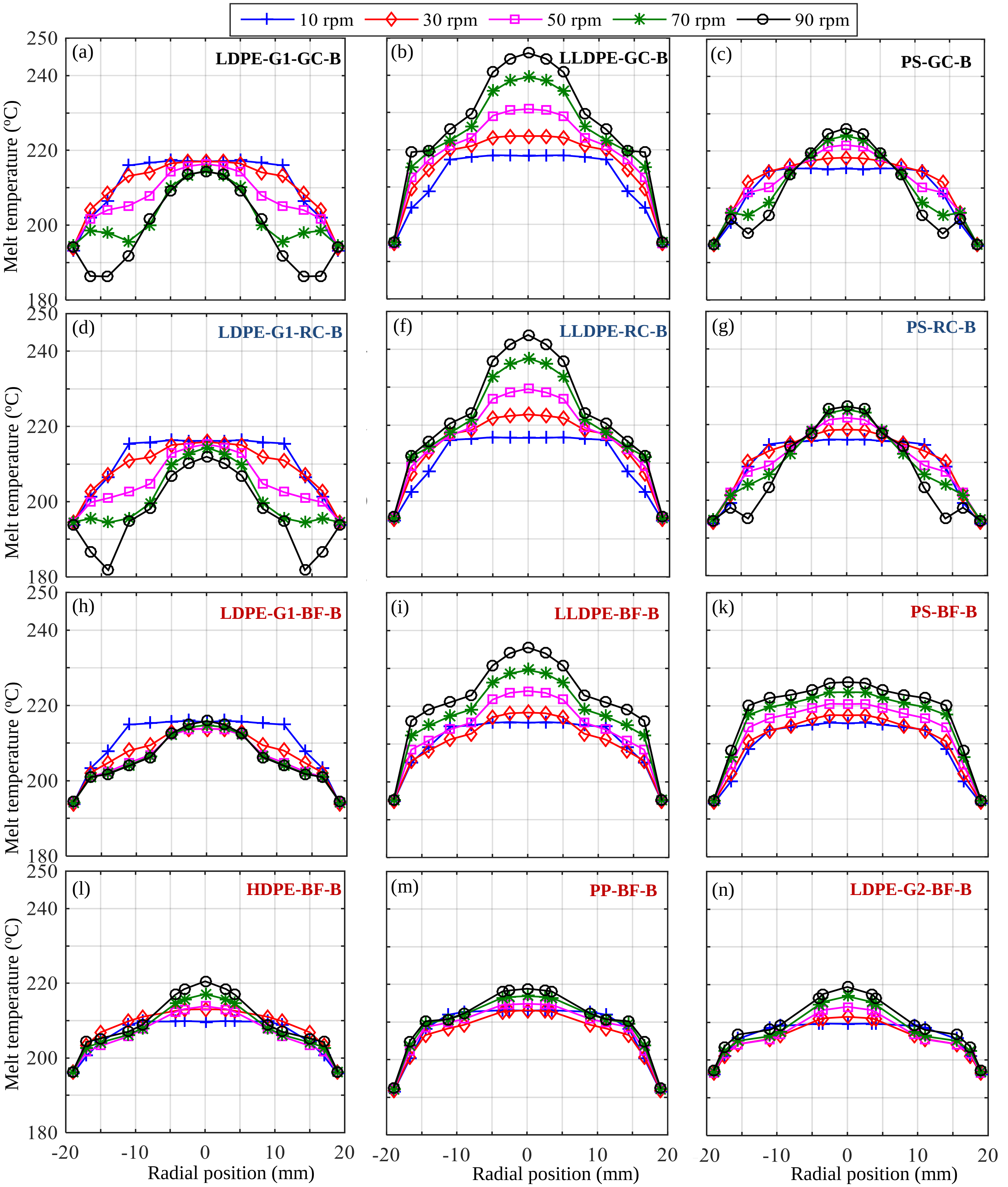

Fig. 3: Melt temperature profiles across the 38 mm diameter die with different materials (LDPE-G1, LDPE-G2, LLDPE, PS, PP, HDPE) with different screw geometries (all sub-figures are at the same scale).

The first 9 sub-figures in Fig. 3 are related to the LDPE-G1, LLDPE and PS materials proceed with all the screws. Sub-figures 3-(a), (b) and (c) present the melt temperature profiles of LDPE-G1, LLDPE and PS with the GC screw under the same processing conditions although the melt temperature profiles are highly variable in nature. Here, LDPE and PS show low temperature shoulder regions at 70 and 90 rpm whereas LLDPE does not. Moreover, significant differences of the magnitude of melt temperature can be noticed with different materials. Generally, for LLDPE melt temperature at all the points increases with screw speed while LDPE-G1 shows an opposite trend. Moreover, PS shows a mixed behaviour where melt temperature at the middles of the flow increases while some other points show a decreasing trend with the increasing speed. Among all the materials, LLDPE shows the highest melt temperature and this is likely a result of the higher shear viscosity and power law index of LLDPE, causing greater shear heating and temperature rise than for the corresponding polymer types. As discussed previously, polymers with a low power-law coefficient such as the LDPE and PS used here (n = 0.39 and 0.28, respectively) will generate less heat by viscous dissipation in the melt with increasing shear rate as screw speed is increased. For example, for LDPE ($n = 0.39$, $K = 6 \times 10^3$) the additional viscous heat generation per unit volume, p, for a 100 s^{-1} increase in shear rate is approximately 3x lower than for the HDPE ($n = 0.41$, $K = 2 \times 10^4$). A measure of the importance of viscous heat generation relative to the heat conduction resulting from the temperature difference between the melt and the walls of the processing equipment (ΔT), is provided by the dimensionless Brinkman number (Br), Eq. (2):

$$Br = \frac{Viscous\,dissipation}{Conduction} = \frac{\eta V^2}{k\Delta T} \tag{2}$$

where V is the average velocity of the flow and k is the thermal conductivity of the polymer. When Br is large (» 1) viscous dissipation is dominant and the temperature profile within the melt will exceed the temperature of the wall. However, if viscous dissipation becomes limited compared to conduction, as is increasingly the case for PS and LDPE as the screw speed is increased, the temperature profile near the wall is increasingly dictated by the temperature of the wall (i.e., conduction) and the influence of the lower temperature settings of the upstream heating zones.

As the same screw geometry and barrel set temperatures were used to process all the materials, the significance of differences in the rheological and thermal properties of each material is clear. Additionally, it has been reported that the polymer form (e.g., pellets, flakes, powder) and pellet shape/size can also affect melt temperature and process thermal behaviour [20-22]. All the materials used in this study were in pellet form but there were slight differences in their shape and size. Therefore, results show that the nature of the melt temperature profiles differs depending on the material and this has also been shown by our previous work [6, 10]. There are shoulder regions, but regions of significantly lower temperature were not observed in the melt temperature profiles of any of the materials as the BF screw was used for all the experimental trials. Furthermore, it is noticeable that the temperature of the melt at the middle of the flow increased with screw speed for all polymer types investigated.

3.3. Effects of processing conditions (screw speed and barrel set temperature)

It is apparent that higher set barrel temperatures resulted in higher mean melt temperature at a particular set screw rotation speed, as was expected from our experience and the results of previous studies [23]. The temperature of the melt in the middle of the flow increased with the screw speed while low temperature shoulder regions (dips) were noticeable at high screw speeds a few millimetres (i.e., 4-8 mm) away from the die wall. However, as shown in Fig. 2, the formation of these shoulder regions clearly depends on the set temperature. For example, with set temperature conditions A and B, no shoulders are visible with the BF screw while a quite small shoulder presents with condition C at 90 rpm. However, these shoulders with relatively large magnitudes can be seen with both GC and BF screws at all the set conditions (A and B: 90 rpm, C: 70 and 90 rpm). For all the screws, the difference between the die set temperature and the lowest temperature at the shoulder region has increased with the set temperature. Moreover, the magnitude of melt temperature fluctuations across the melt flow (i.e., the difference between the lowest and the highest melt temperature across the melt flow) is highly dependent upon both the screw speed and set temperature and these fluctuations tend to increase as these two parameters are raised. Differences in the shape of the melt temperature profiles can be significant even at the same processing conditions (i.e., same set temperature and screw speed) for different materials and screw geometries as evident from Fig. 3 and similar observations have been reported in the literature [24]. Overall, these results emphasize the importance of the selection of appropriate process settings (i.e., the optimum process operating point) to achieve the desired melt quality for a given machine and material.

As was discussed above, the formation of the low temperature shoulder region depends on the materials being processed, screw geometry and the set conditions used during processing. Moreover, it is well-known that the material, machine and process parameters in polymer extrusion are heavily coupled in nature [4-13, 25-27]. Hence, understanding of this phenomenon is quite complicated and hence further research is highly recommended. Overall, the results of this study also highlight the complexity of the melt flow behaviour in polymer processing and hence further research should be focused on expanding the understanding in this area.

4. Conclusions

Detailed information on the melt homogeneity of a single screw extruder was obtained using a thermocouple mesh sensor. Process thermal homogeneity was significantly affected by screw geometry, material properties and process settings; and melt temperature was found to be highly variable under different processing conditions. The relationship between polymer type, extruder screw geometry and process settings was found to be complex in nature, leading to a highly variable and unpredictable process. The effect of process settings and screw geometry on melt temperature homogeneity is influenced by factors such as heat generation by conduction and viscous shear, mixing and residence time. The six different polymeric materials used in this study showed considerably different thermal behaviour with different screw geometries, even with the same process settings. These observations emphasize the importance of appropriate selection of screw geometry and set extrusion conditions for a given polymer to avoid unnecessary thermal fluctuations which can be detrimental to product quality. These results show the melt temperature to vary significantly across the melt flow cross-section, and this type of detailed information cannot be obtained from single point measurement techniques commonly used in the polymer processing industry. Thermal profile measurement techniques are not yet industrially available and therefore it is desirable to continue development of suitable robust and non-intrusive techniques.

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