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C.-C. Cheng^{1,*}, H. Banakar², B.-T. Ooi², C.-K. Jen³¹Dept. of Electrical Engineering, Hsiuping Institute of Technology, Da-Li, Taichung, Taiwan²Dept. of Electrical and Computer Engineering, McGill University, Montreal, Quebec, Canada³Industrial Materials Institute, National Research Council Canada, Boucherville, Quebec, Canada

Melting Quality of Polymers in Internal Mixer Diagnosed by Ultrasound

Real-time, non-intrusive and non-destructive ultrasonic technology has been used to monitor the melting process in an internal mixer. Visual observation, mechanical torque measurement, and ultrasonic signatures, such as amplitude and time delay of transmission and reflection echoes were used for the diagnosis of the melting process of low density polyethylene (LDPE). Phenomena during the melting process, including phase change from solid to melt, partially melted pellets, air bubbles inside the melt, were successfully monitored by ultrasound. The ultrasonic signatures were able to determine when the polymer has melted completely. The method of moving standard deviation (MSD) was applied to establish the melting completion timing accurately. Higher temperature of mixing chamber and faster rotation speed of blades reduced melting completion period, indicated by MSD of ultrasonic signatures. The presented ultrasonic technique can be utilized to optimize the melting process.

1 Introduction

The internal mixer is one of the essential instruments widely used in most laboratories working on polymer or powder material and processing. It requires only a small amount of materials and is usually used as a preliminary testing step for verifying the melting and mixing behaviors of polymers, chemical reactions in polymers, polymer cross-linking and polymer degradation before the actual processing in extruder. Internal mixers are usually equipped with a torque meter. Due to the small dimensions of the mixing chamber (bowl), torque data measured by the torque meter are reasonably representative of the actual torque exerted on the polymer melt. Based on the measured torque, four distinct rheological phases, during the melting process of polymer, may be identified (Shih et al., 1991). They are: (1) elastic solid pellets; (2) deformable solid pellets; (3) transition materials; and (4) viscoelastic fluid. The processes include complex elastic and viscous phenomena of polymers.

* Mail address: Chin-Chi Cheng, Dept. of Electrical Engineering, Hsiuping Institute of Technology, 11, Gongye Rd., Da-Li City, Taichung, Taiwan, 412-80
E-mail: newmanch@mail.hit.edu.tw

When polymer pellets become viscoelastic fluid, the torque reaches a steady state. The time required for torque to reach the steady state is usually seen as melting completion timing, and it depends on the material and the processing conditions (temperature and rotor speed, etc.).

Many researches have used internal mixer data to find physical and chemical phenomena related to melting, mixing and reaction processes. Through numerical simulation, Jongen (2000) and Ghoreishy et al. (1997) had demonstrated that different geometry or shape of chamber and blades affect the efficiency of the process. Zalc et al. (2002) suggested that the lower flow rates produced more effective mixing at a smaller energy cost in static mixers. Goodrich and Porter (1967), Serpe et al. (1990), and Bousmina et al. (1999) developed models and experimental procedures for indirect or direct estimation of shear rate and viscosity using internal mixer rotor speed and torque data. However, little effort had been directed at developing online techniques and methodologies for determining the melting and mixing quality in order to optimize the process.

Ultrasonic technique is one of the candidates for monitoring the different melting stages during process, because its basic signatures, such as propagation characteristics (velocity and attenuation) (Tatibouet and Piche, 1991; Gendron et al., 1995), reflection (Wang et al., 1997; Cheng et al., 2007) and transmission coefficients (Michaeli and Starke, 2005) and scattering signals in materials, are associated with physical and rheological properties of materials (Tatibouet and Piche, 1991; Gendron et al., 1995), as well as process dynamics, process parameters and product qualities (Wang et al., 1997; Cheng et al., 2007; Michaeli and Starke, 2005; Brown et al., 1999). To further demonstrate this, a research applying the ultrasonic delay line probe for process diagnosis in the internal mixer is initiated. The objective of this study is to investigate ultrasonic techniques for melting process diagnosis in order to improve productivity and efficiency.

2 Experiments

The internal mixer is equipped with a mixing chamber and two rotating blades with roller shape, which replicates the shape of the screw in the extruder, as shown in Fig. 1. The mixing chamber has two cylindrical cavities for melting and mixing pro-

Fig. 1. Mixing chamber with rotating blades, thermocouple and ultrasonic delay line probe

cesses. The cavity has a diameter of 39.8 mm and a depth of 48.5 mm. These two cavities have the capacity of 60 ml, with two roller blades. There are heating coils surrounding the mixing chamber. The internal mixer can operate up to 300 °C and rotate up to 108 rotation per minute (min^{-1}). The testing material is fed into the mixing chamber from a feed window in the top. The dimensions of the head of the rotating blade are 36.7 mm in diameter and 47 mm in length. As shown in Fig. 1, the total lengths of rotating blade 1 and 2 are 185 mm and 156 mm, respectively. The blade 1 is directly fitted on the rotor shaft, while blade 2 is geared to the shaft. The mixing chamber, rotation blades, thermocouple and ultrasonic delay line probe are installed into the internal mixer machine for diagnosis of melting process.

An ultrasonic delay line probe (Jen et al., 2000) is installed into the mixing chamber of internal mixer (C. W. Brabender 2000) for real-time online melting process diagnosis, as shown in Fig. 2. The central frequency of the employed commercial available ultrasonic transducer (UT) is 10 MHz. The ultrasonic probe has the compatible geometry and interfaces with those of conventional temperature and cavity pressure sensors (for example, TPT 467XL, Dynisco Instruments & Polymer Test, Franklin, MA). Therefore, it can be installed into the internal mixer machine without modification of the existing chamber. It also means that temperature, pressure and ultrasonic sensors are interchangeable. The ultrasonic delay line probe can operate up to 600 °C, which is sufficiently high to encompass the full range of melt temperatures typically encountered in polymer processing. The UT end of the probe is cooled down by the compressed air to a temperature lower than 40 °C, which is

Fig. 2. Ultrasonic delay line probe with cooling section and UT

Fig. 3. Schematic drawing of a cross-section of mixing chamber, including ultrasonic delay line probe, rotating blades and thermocouple, presents ultrasonic paths in probe and polymer

required to operate such UT. By designing the shape and size of probe, the cooling effect at the UT end of the probe had been considered so that the temperature of the melt to be monitored will not be affected. Therefore, the monitoring of the melting behavior at the probing end was not affected by the cooling section at the UT end of the probe.

A schematic drawing of a cross-section of the mixing chamber with the ultrasonic delay line probe, rotating blades and thermocouple, is given in Fig. 3. It illustrates the paths of ultrasound propagating in the probe and the polymer melt. The probing end faces the rotating blade 1. The first round trip ultrasonic longitudinal-wave echo, indicated by L^1 , is reflected at the probe end/air or polymer interface, depending on whether the polymer is attached to the inside surface of mixing chamber or not. The n -th round trip echo from the flight of the rotating blade half through the polymer melt and reach back to UT is L_{2nf} ($n = 1, 2, \dots$).

The internal mixer is also equipped with a torque meter, attached to the rotator of the driving motor, and a K-type thermal couple, located at 0.2 mm depth from the inside surface of mixing chamber bottom, as shown in Fig. 3. The torque and temperature data, acquired during the entire melting cycle, are used for comparison with ultrasonic data for all experiments.

The polymer employed is a low-density polyethylene (LDPE) (from Nova Chem., Alberta, Canada). The density is 0.926 g/cm^3 provided by the company. The maximum capacity of the mixing chamber with the roller blades is 56 g of LDPE in the experiments. The procedures of melting process are as follows: First, the empty mixing chamber is heated up to the setting temperature and the blades are rotating at the desired speed. Then the polymer pellets, 50 g, are fed into the cavity for melt. The melting conditions employed in the experiments are as follows: the setting temperatures of the mixing chamber are 170, 200 and 230 °C; the rotation speeds of blades are 14, 28 and 42 min^{-1} .

3 Results

Fig. 4 shows the amplitude variations of torque and temperature during the melting process of 50 g LDPE under the tem-

Fig. 4. Variation of torque and temperature during melting process of 50 g LDPE. Temperature: 200°C and rotation speed: 28 min⁻¹

perature setting of 200°C and rotation speed of 28 min⁻¹. In the process time of 30 s, 50 g of LDPE were fed into the mixing chamber. From process time of 30 to 50 s, the torque value increased due to the friction, and temperature decreased due to the heating transfer between polymer pellets, rotating blades and mixing chamber. At process time of 50 s and 52 s, the torque and temperature reached to their peak and bottom values, respectively. After that, the torque decreased due to the reduction of viscosity of the polymer melt, and temperature increased due to the heat coming from the heating coils, friction and shear forces exceeding that absorbed by the polymer. At process time of 210 s, the melting process stopped since the torque reached a steady state and the temperature almost recovered its original value.

Fig. 5A to F are photographs of the melting process in Fig. 4 at the process time of 37, 50, 64, 79, 121, and 200 s, respectively, taken from the feed window. In the beginning, the mixing chamber was empty. At process time of 30 s, the polymer pellets were fed into the mixing chamber. In Fig. 5A, at the process time of 37 s, which was 7 s after polymer fed into chamber, some of the pellets were still in the feed window and not entered into the mixing chamber yet. In Fig. 5B, at process time of 50 s, which was the time that torque reached its peak value, most of the pellets were in the elastic solid form and bound with rotating blade. Few pellets were melted and attached to the surface of the mixing chamber. In Fig. 5C, at process time of 64 s, which was 14 s after the peak value of torque, most of the solid pellets had been melted and were dispersed in the mixing chamber. But some of partially melted pellets still existed within the melt, as indicated by dashed circles.

In Fig. 5D to F, at the process time from 79 to 200 s, even though the torque and temperature still varied, there were no solid or partially melted pellets left and the polymer melt didn't appear to change significantly. This may indicate that, after the process time of 79 s, most pellets were already melted. In Fig. 5E and F, the bubble shape material shown in the left side of figure was the polymer melt brimming out of the chamber. This may be due to density reduction at higher temperature, and it was confirmed from the Pressure-Volume-Temperature (PVT) measurement (Piche et al., 1988). It was our interest to diagnose the dynamic melting process after most pellets having been melted. In order to feed additional materials into the mixing chamber, the amount of LDPE

Fig. 5. Melting process from feed window in Fig. 4 at process time of 37, 50, 64, 79, 121 and 200 s

should be reduced to 40 g to avoid the polymer melt brimming out of the chamber. The melting phenomena in Fig. 5 were expected to happen early when the amount of material was 40 g.

Fig. 6 shows the trace of ultrasonic waveforms acquired with the ultrasonic probe in Figs. 2 and 3 during the process time when polymer had melted. Ultrasonic signals were ac-

Fig. 6. Sample traces of ultrasonic waveforms showing delay times between L^1 and L_{2f} and between L_{2f} and L_{4f} echoes

quired every 8.3 ms (i.e. 120 Hz) during the entire cycle. However, a process time interval of 16.7 ms (i.e. 60 Hz) had been selected for clarity and was shown in Fig. 6 for a period from 83.90 to 84.10 s. The L^1 , propagating in the delay line probe and reflected at the probing end/polymer melt interface, was observed throughout the period. When the flight of the rotating blade arrived below the probe location, the L_{2f} and L_{4f} echoes appeared from the process time of 83.94 to 84.04 s, at time delay of 50.4 μ s and 53 μ s, respectively. The L_{2f} and L_{4f} echoes existed while the top surface of the flight was facing the probe location. Echoes reflected from the other two flights were in the similar waveforms. For the data shown in Fig. 6, the time delay Δt between the L^1 and L_{2f} echoes, or L_{2f} and L_{4f} echoes, is directly related to ultrasonic velocity in the polymer and indicates the phase and melting condition of the polymer inside the mixing chamber (Sun et al., 2004a; 2004b). The ultrasonic velocity decreases, or the time delay increases, as the polymer melts.

Fig. 7 presents the amplitudes of the L_{2f} echoes from three flights of the rotating blade 1 with respect to the process time, when the polymer was melted. It is noted that the processing period here is different from that in Fig. 6. In Fig. 7, at process times around 75.6, 76.3 and 76.9 s, peaks of the L_{2f} echoes were observed, indicating the arrival of each flight of the blade 1 directly beneath the probe location. The peak amplitudes of three L_{2f} echoes varied, due to different ultrasonic reflecting areas of the three flights. The three flights of blade 1 did not have the identical shapes, as shown in Fig. 2. The duration of each pulse was around 0.1 s. The peak amplitude of L_{2f} echo and the time delay between the L^1 and the L_{2f} echoes were used as the indicator for the melting quality. This will be discussed further in section 4.1. In the case of resin with a large damping ratio, the reflected ultrasonic echo going through the melt would be affected. This may limit the application of this method. However, the ultrasonic frequency can be lowered for the inspection of resins with a large damping ratio. Once the reflected echoes can be observed, the diagnostic technique will be feasible.

Fig. 7. Amplitude of L_{2f} echoes of flights 1, 2 and 3 of rotating blade 1 (see Fig. 3) in process time after the polymer is melted

4 Discussions

4.1 Melting Completion

Determination of the melting completion using ultrasound would give an opportunity to end the melting process earlier than the conventional method, which requires the torque reaching a steady state and the temperature recovering its initial value. In this experiment, 40 g of LDPE, as the host material, were fed into mixing chamber for melt first, and, after the previous polymer had been melted completely, additional 4 g of same material were fed for diagnosing dynamic melting process. Fig. 8A and B present the amplitudes variation of torque and temperature acquired by the torque meter and thermal couple. Fig. 8C, D and E show the amplitude variation of L^1 echo, peak value of L_{2f} echo and the time delay between L^1 and L_{2f} echoes, respectively, during the melting process. The time delay between L^1 and L_{2f} echoes and the peak value of L_{2f} echo are shown in Figs. 6 and 7, respectively. The amplitude of L^1 echo is normalized by its maximum value. It is noted that the presented peak amplitude of L_{2f} echo and the time delay between L^1 and L_{2f} echoes, are from the same flight of the blade 1. The temperature setting of the mixing chamber and the rotation speed of the blade are 200 °C and 28 min⁻¹, respectively.

Before the process time of 30 s, the mixing chamber was heated up to 188 °C and the blade rotated at the speed of 28 min⁻¹. It is noted that even under the temperature setting of

Fig. 8. Variation of (A) torque, (B) temperature, (C) amplitude of L^1 , (D) peak value of L_{2f} , and (E) time delay between L^1 and L_{2f} echoes during melting process. LDPE introduced: 40 g at 30 s; 4 g at 150 s

200 °C, the actual chamber temperature was 188 °C, due to the heating loss from the feed window. At the process time of 30 s, since the host material (40 g LDPE) was fed into the mixing chamber, the torque value rose up quickly. At the process times of 31.6 s and 32 s, the amplitude of L^1 echo and temperature started to decrease, respectively. It is noted that there was a time difference between torque, temperature and ultrasonic measurement. This is due to the fact that the torque value reflects an overall or an “integrated” material state, while the temperature and ultrasonic measurements detect local conditions. However, when the roller rotates one turn, many of the melting/mixing states of the whole samples will be observed by ultrasonic delay-line probe. The torque and temperature values varied as described previously. The drop in the value of L^1 echo was due to the fact that, since some LDPE pellets were already melted and they got attached to the inner surface of mixing chamber, part of the ultrasonic energy was transmitted into the polymer melt through the probe end/polymer melt interface. The fluctuation of L^1 echo indicated the random contact of soft pellets and molten polymer to the probing end surface.

At the process time of 44 s and 48 s, the torque and temperature value reached the peak and bottom value of 14.8 Nm and 154.9 °C, respectively. Around this time, the amplitude of L^1 echo also reached its lower level, but kept fluctuating. According to Fig. 5B, during this period, most of the pellets were still solid with partially melted. At the process time of 45 s, the ultrasonic echo L_{2f} reflecting from the polymer melt/flight of the blade 1 interface started to appear. In the process time from 45 to 73.4 s, the peak amplitude of L_{2f} echo and the time delay between L^1 and L_{2f} echoes fluctuated, indicating the non-uniformity of the melting condition. This was also evident from Fig. 5C. Except one peak occurring at the process time of 68 s, fluctuations of the amplitude of L^1 stopped at the process time of 51 s. This peak may result from a partially melt pellet. After the process time of 73.4 s, the peak amplitude of L_{2f} echo reached its upper level, and the time delay between L^1 and L_{2f} echoes entered into a stable state, indicating that there was no solid or partially melted pellets and all the LDPE pellets were melted. Fig. 5 also supported those diagnosed by ultrasonic technique. After this moment, the increase of time delay between L^1 and L_{2f} echoes from 73.4 s to 150 s in Fig. 8E was due to the increase of melt temperature. Therefore, when the amplitude of L_{2f} echo reached its upper stable level and the time delay between L^1 and L_{2f} echoes became stable, all polymer pellets had completely melted and further heating or rotating may not be necessary. The information of melting completion may be useful to optimize the process. However, the torque and temperature values provided such information later.

At process times 84 s, 120 to 130 s and 139 s, the amplitude of L_{2f} drop slightly. This may be due to air bubbles introduced between the probe and blade surface resulting from insufficient material. This phenomenon will be further discussed in section 4.2. However, air bubbles didn't alter the time delay between L^1 and L_{2f} echoes significantly.

In order to diagnose the dynamic melting process, at process time of 150 s, a 4 g LDPE was fed into the mixing chamber. The reactions of these five parameters were similar with those of host material. At process time of 160.6 s, the peak amplitude of L_{2f} echo recovered its original value and the time delay between L^1 and L_{2f} echoes stopped fluctuating, indicating com-

plete melt of the additional material. These results present that ultrasonic signatures are able to diagnose the dynamic melting process. Since the dynamic melting phenomena of additional material were similar with the host material, rest of the experiments would focus on the 40 g host material only.

In order to capture the timing of melting completion through the ultrasonic signals, moving standard deviations (MSDs) of the ultrasonic parameters are calculated. The MSD is a statistic calculating and analyzing method, through which the variation of system can be captured, when the system structure changes or noise or perturbation are added to the system. MSD has been used to predict the stock prices on the stock markets (see Appendix). Fig. 9A, B and C show the MSD values for a window size of 3 points apply to the amplitude of L^1 echo, peak amplitude of L_{2f} echo and time delay between L^1 and L_{2f} echoes, respectively. In Fig. 9A, after the host material of 40 g LDPE was fed into the mixing chamber, the MSD of L^1 echo rapidly increased to 0.062 at the process time of 31.6 s, and then decreased gradually to noise level at the process time of 50 s, except one peak at the process time of 68.5 s. In Fig. 9B and C, the MSDs of peak amplitude of L_{2f} echo, and time delay between L^1 and L_{2f} echoes started to appear and increase at the same process time of 49.3 s, and decreased to noise level at process time of 75.6 s and 77.3 s, respectively. According to Fig. 5D, the timing when the MSDs of peak amplitude of L_{2f} echo and time delay between L^1 and L_{2f} echoes reached the

Fig. 9. Moving standard deviation (MSD) of (A) amplitude of L^1 , (B) peak amplitude of L_{2f} and (C) time delay between L^1 and L_{2f} echoes. Window size of MSD is 3 sample points

noise level could be used to indicate the complete melting point. The period, when the host material was fed into the mixing chamber until it reached its melting completion, was defined as the melting period for host material.

At process time of 150 s, the additional material of 4 g LDPE was fed into the mixing chamber, causing the MSDs of three parameters to rise simultaneously. At process time of 155 s, the MSD of amplitude of L^1 echo reached to the noise level. At process time of 162.7 s and 164.8 s, the MSDs of time delay between L^1 and L_{2f} echoes and peak value of L_{2f} echo reached to the noise level, respectively, indicating that the impulse material was melted completely. Therefore, the presented ultrasonic technique produces a measurement of melting completion, which can be used to optimize the process parameters.

4.2 Air Bubble Effect

To prove our assertion concerning the effect of air bubbles on the peak amplitude of ultrasonic L_{2f} echo in the section 4.1, two simple experiments were carried out. The settings of the temperature of the mixing chamber and the rotation speed of the blades were 200°C and 28 min^{-1} , respectively. The amounts of material for these two experiments were 30 g and 50 g of LDPE, representing insufficient and full amount of material, respectively. Fig. 10A and B show the results of the peak amplitude of L_{2f} echo for these two experiments during the melting process. In Fig. 10A, the peak amplitude of L_{2f} echo reached the upper level at process time of 71.7 s and kept in the stable state until 80.3 s. From the process time of 80.3 to 150 s, air bubbles altered the peak amplitude of L_{2f} echo around 11 times. The drop of peak amplitude of L_{2f} echo in Fig. 10A was deeper than those in Fig. 8D. This may indicate that the air bubbles in the 30 g experiment were larger and more than those in the 40 g experiment. In this experiment, the size of air bubble was not easy to measure. However, from our other experiment, even small size of particles can be detected by ultrasonic probe. The influence of filler may affect the detection of air bubble. This needs further investigate in

Fig. 10. Peak amplitude of L_{2f} echo. (A) 30 g and (B) 50 g LDPE

Fig. 11. Time delay between L^1 and L_{2f} echoes. (A) 30 g and (B) 50 g LDPE

the future. In Fig. 10B, the peak amplitude of L_{2f} echo reached the upper level at the process time of 78.6 s and kept in the stable state until 150 s. Air bubbles did not alter the peak amplitude of L_{2f} echo in Fig. 10B during the melting process, because the presence of full material in the mixing chamber minimized the creation of air bubbles.

Fig. 11A to B show the results of time delay between L^1 and L_{2f} echoes, which also indicate the same phenomena as those in Fig. 10A and B. The time delay value in Fig. 11A was lower than that in Fig. 11B, indicating the higher melt temperature in Fig. 11A due to fewer polymer pellets involved. Because the time delay between L^1 and L_{2f} echoes was more stable than peak amplitude of L_{2f} echo, the MSD of time delay between L^1 and L_{2f} echoes would be used as an indicator for melting completion in the following sections. These two experiments also show that more material needs longer time to melt completely. Our other experiments also demonstrate that faster rotation speeds would introduce more air bubbles.

4.3 Temperature Effect

The temperature of mixing chamber affects the melting process significantly. Higher temperature settings help polymer melt faster, but also cause faster material degradation. Therefore, choosing a proper temperature setting is critical for increasing melting efficiency and optimizing the process. In order to investigate the temperature effect on the melting process, the temperatures of the mixing chamber were set at 170 , 200 and 230°C , separately, to melt 40 g LDPE with a fixed rotation speed of 28 min^{-1} . The experimental results of torque and MSD of time delay between L^1 and L_{2f} echoes with respect to process time are presented in Fig. 12A to D. In Fig. 12A, the melting process with lower temperature setting required higher torque values and needed more time to reach the steady state. In Fig. 12B to D, the MSD of time delay between L^1 and L_{2f} echoes reached the noise level earlier, when the melting process was set at a higher temperature. The timings of melting completion were at process times of 102.7, 84.0 and 66.8 s for

Fig. 12. Effect of temperature setting on melting process: (A) torque measurements, (B), (C) and (D) MSD of time delay between L^1 and L_{2f} echoes. Rotation speed: 28 min^{-1} ; load: 40 g LDPE

the temperature settings of mixing chamber at 170, 200 and 230°C , respectively.

In order to view clearly the temperature effect, the melting period and peak torque value are compared with the temperature settings of the mixing chamber. The results are shown in Fig. 13. In the temperature range from 170 to 230°C , the melting period decreased from 72.7 to 36.8 s and the peak torque value decreased from 15.8 to 12.96 Nm. This indicates that the higher temperature setting of mixing chamber would cause lower torque response and reduce the melting period. The ul-

Fig. 13. Variation of melting period and peak torque with temperature setting of mixing chamber

trasonic technique can clearly indicate the melting completion at each temperature setting, while the torque and temperature techniques do not.

4.4 Rotation Speed Effect

It may appear that the rotation speed of the blade will affect the melting process as much as the temperature of the mixing chamber. In order to confirm this, the rotation speeds of the roller blade were set as 14, 28 and 42 min^{-1} , to melt 40 g LDPE with the fixed temperature setting of mixing chamber of 200°C . The experimental results of the torque and MSD of time delay between L^1 and L_{2f} echoes with respect to the process time are presented in Fig. 14A to D. In Fig. 14A, the melting process with lower rotation speed setting indicated lower torque value and need more time to reach a steady state. In Fig. 14B to D, the MSD of time delay between L^1 and L_{2f} echoes reached the noise level earlier, when the melting process was set at the higher rotation speed. The complete melting timings were at process time of 84.7, 84 and 74 s for the rotation speed setting of 14, 28 and 42 min^{-1} , respectively.

In order to view clearly the rotation speed effect, the melting period and peak torque value are examined for different rotation speed settings. The results are shown in Fig. 15. In the rotation speed range from 14 to 42 min^{-1} , the melting period decreased from 54.7 to 44 s and the peak torque value increased

Fig. 14. Effect of rotation speed on melting process: (A) torque measurements, (B), (C) and (D) MSD of time delay between L^1 and L_{2f} echoes. Temperature setting: 200°C ; load: 40 g LDPE

Fig. 15. Variation of melting period and peak torque with rotation speed

from 11.4 to 15 Nm monotonically. This indicates that faster rotation speed would cause higher torque response, which called for higher mechanical force, but reduced the melting period. Again, the ultrasonic technique can clearly indicate the melting completion, while the torque and temperature techniques do not.

5 Conclusions

Real-time, non-intrusive and non-destructive monitoring of melting process in the internal mixer has been performed using ultrasonic delay line probe. The melting process of 40 g LDPE from solid pellets to polymer melt was diagnosed by visual observation, torque, temperature, and ultrasonic signatures. The machine settings were 200 °C of temperature of mixing chamber and 28 min⁻¹ of rotation speed of blades. The timing of melting completion can be clearly determined by MSD of the ultrasonic time delay. The partially melted pellets and air bubbles during melting process can also be detected. The melting period is reduced when the temperature of mixing chamber or rotation speed of the blade increases. Therefore, the presented ultrasonic techniques may be an optional approach to diagnose melting processes nondestructively and noninvasive, in order to optimize the process parameters, reduce cost and evaluate melt quality.

Appendix: Moving Standard Deviation (MSD)

General

In the field of probability and statistics, the method of standard deviation (SD) is used to calculate the spread of values of probability distribution, random variable, or population, relative to the expected values (Hayslett, 1968). SD is the most common measure of statistical dispersion or variation, evaluating how widely values in a data set are spread. A higher SD means that data points are far from mean value and vice versa. The method of SD can be used to set bounds on deviations of future values from their predictions, which are calculated from the past information.

Moving Standard Deviation (MSD) is an extension of SD applicable to online systems. MSD can capture system structure in the presence of noise and input changes. It has been used as a tool to analyze the nature of changes occurring in stock prices on the Stock Markets. In this paper, the concept of MSD has been deployed to identify transitions in the polymer state; specifically, the time of melting completion. Such transitions are accompanied with changes in the parameters that characterize the polymer, making MSD the appropriate online tool to detect the on-set and the completion of such changes. To the authors' knowledge, MSD has been rarely used in process control systems and it may be the first time to be used in the work reported in this paper (Qin and Badgwell, 1997).

MSD Calculation

For a process variable X , the MSD is defined based on a time window containing the N past samples of X , ending with the most recent sample. The N samples in this window are used to calculate the MSD of X according to,

$$\text{MSD} = \sqrt{\frac{1}{N} \sum_{i=1}^N (X_i - \bar{X}_w)^2}, \quad (1)$$

where, $\bar{X}_w$ is the window's Moving Expected Value (MEV), given by,

$$\bar{X}_w = \frac{1}{N} \sum_{i=1}^N X_i. \quad (2)$$

On arrival of a new sample, the window is shifted forward in time by one sampling period and the MSD is recalculated. The forward shift of the window is accomplished by simply dropping the first sample in the window and adding the most recent sample to it. This procedure is continued in time, creating a time series for MSD. Monitoring this time series can provide information on changes in either the process noise level or process structural changes.

The choice of N (i.e. the length of the window) and, therefore, the sampling rate, are critical to the quality of the information generated by MSD. These quantities have to be chosen carefully, making sure that they are suitable for the time constant associated with the phenomenon to be captured.

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