

IN-LINE ULTRASONIC MONITORING OF POLYMER BLENDING IN A TWIN-SCREW EXTRUDER

Z. Sun¹, J. Tatibouët¹, C.-K. Jen¹, H. L. Liang², and C.-Y. Su²

¹ Industrial Materials Institute, National Research Council Canada, Boucherville, Quebec, Canada;

² Mechanical and Industrial Engineering Department, Concordia University, , Montreal, Quebec, Canada

Abstract: Polymer blending using a twin-screw extruder is a widely used process in polymer processing industry by which more than one polymers are blended under molten state to achieve properties that are not available with each of the individual polymer. In the current work, high temperature ultrasonic sensors have been installed at several locations on a twin-screw extruder to monitor the blending process at various locations along the polymer flow direction inside the mixing channel of the extruder and at the exit of the extruder. The polymer blends were composed of a high-density polyethylene (HDPE) and a polystyrene (PS). The experiments were carried out under different mixing intensities, feed rates, and PS concentrations. Samples were collected at ultrasonically probed locations and then compared with the ultrasonic measurement data. The study has revealed close relations between the ultrasonic data and the status of the polymer blends and demonstrated that the ultrasonic technology presented in this communication can be a valuable tool for providing in-process information about polymer blending processes.

Introduction: Polymer blending using a twin-screw extruder is a widely used process in polymer processing industry by which more than one polymers are blended under molten state to achieve properties that are not available with each of the individual polymer. The quality of polymer blending, which is characterized by the degree of dispersion of a minor phase polymer into the major phase polymer, has great effect on the quality of the final product. The improvement and control of the quality of polymer blending through extruder and process designs and on-line or off-line process monitoring have been and continue to be important R&D activities in the plastics industry. There have been published work on the use of ultrasound for monitoring polymer blending and the dispersion of a mineral filler in a polymer matrix during extrusion.¹⁻³ In these researches, the monitoring was carried out either at the die¹ or in the extruder barrel²⁻³, and the fluctuation of ultrasonic signal strength has been used to infer the state of dispersion. It has also been shown that the ultrasonic velocity in a polymer blend can provide information about the blend composition and the ultrasonic attenuation in the blend can provide information about blend phases morphology.⁴ The current work was one of the continuing efforts in furthering the understanding of the interaction between ultrasound and polymer blends through experiments under various conditions and comparisons between ultrasonic data and the morphology of the samples collected at the ultrasonically monitored locations. The objective of the research is to develop an in-line ultrasonic technique capable of providing real-time information on the progress of polymer blending inside the extruder mixing channel.

Experimental setup: A Leistritz 34-mm twin-screw extruder was used. A schematic layout of the extruder is illustrated in Figure 1(a). This extruder has a mixing chamber composed of 12 heated barrels and two 34-mm diameter co-rotating mixing screws inside the chamber. Three ultrasonic clad buffer rod probes, denoted as Probes I, II, and III respectively, were mounted at the melting, mixing, and pumping zones of the extruder (Fig. 1(a), (c), and (d)). The probing ends of the probes, which had the same external diameter and thread as conventional Dynisco pressure and temperature probes, were flush with the internal surface of the barrel wall with probe axis perpendicular to the screw shaft and pointing to the center of the latter. The other ends of the probes to which a 5-MHz longitudinal piezoelectric ultrasonic transducer was attached, were cooled with compressed air to maintain its temperature below 40 °C. Probes I, II, and III sent ultrasonic pulses to the polymer. The ultrasonic signals were then reflected by the rotating screw and the reflected signals were acquired at a 100-MHz sampling rate by a data acquisition system. An instrumented die was installed at the exit of the extruder (Fig. 1(a) and (b)). Two ultrasonic buffer rod probes were mounted opposite each other on the instrumented die. These two probes operated in transmission mode and were cooled with water.

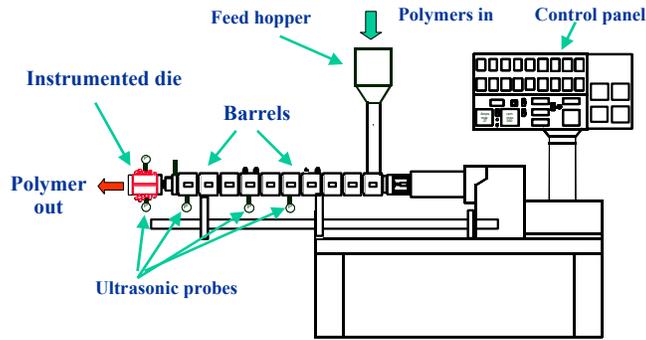


Fig. 1(a)



Fig. 1(b)

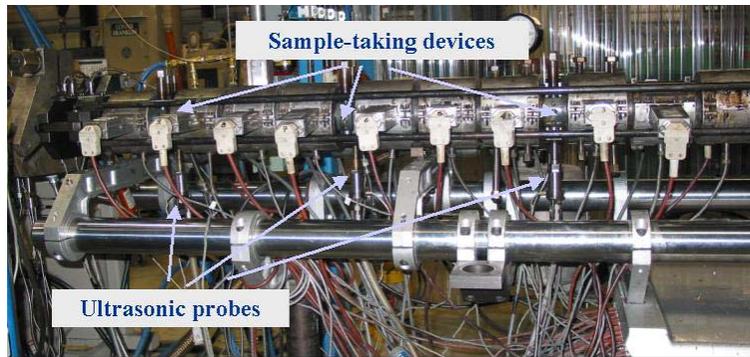


Fig. 1(c)

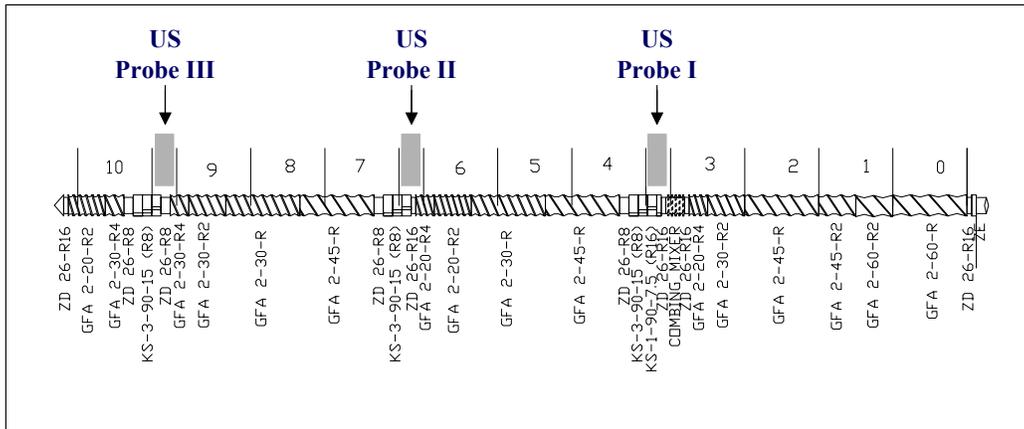


Fig. 1(d)

Figure 1. Ultrasonic in-line monitoring of polymer blending on a Leistritz 34-mm twin-screw extruder. (a) Schematic layout of the extruder; (b) two ultrasonic probes mounted on an instrumented die; (c) external view of three ultrasonic probes mounted at the melting, mixing, and pumping zones of the extruder and three sample-taking devices mounted at the same locations as the ultrasonic probes; (d) configuration of the screw used in this study. The areas monitored by the ultrasonic probes are indicated with arrows.

The extruded materials were blends of a high-density polyethylene (HDPE) and a polystyrene. The screw rotation speed was 200 RPM, and the barrel temperature was 200 °C at all sections. Monitoring was carried out for two feed rates (7 kg/h, and 10 kg/h) and four PS concentration levels (0, 2, 4, and 8 weight %). Samples were collected at the ultrasonic probes locations with the help of specially designed sample-taking devices.

Measurement principle: Polymer blending in a twin-screw extruder is mainly achieved via shear forces exerted on the polymers by the rotating mixing elements (screws). As the molten polymers are flowing from the feed entrance to the extruder exit, the dispersion of the minor phase polymer into the major phase polymer is expected to become better and better (Fig. 2). This change in polymer morphology is expected to result in a change of ultrasonic attenuation in the polymer blend. In other words, ultrasonic attenuation could provide information about the progress of polymer blending along the polymer melt flow direction inside the mixing channel.

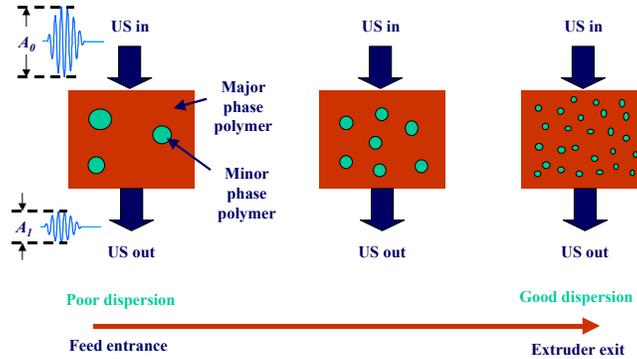


Figure 2. Ultrasonic attenuation is expected to be sensitive to the degree of dispersion of the minor phase polymer into the major phase polymer. In the figure, “US in” and “US out” denote the ultrasonic waves going in and coming out of the polymer blend, respectively, and A_0 and A_1 represent the peak-to-peak amplitudes of the in and out waves. The difference between the amplitudes A_0 and A_1 accounts for the attenuation of ultrasonic energy in the polymer blend.

Results and Discussions: Figure 3 displays the ultrasonic echo signals reflected by the screw flights at Probe I location during extrusion of the pure HDPE and during about two screw revolutions. The first echo is the one that has made one round trip between the screw flight tip and the ultrasonic probe of which the probing end was flush with the internal wall of the barrel. Every time a screw flight tip was crossing the probed area, an ultrasonic echo was reflected by the flight tip and received by the probe. The second echo indicated in the figure is the one that has made two round trips between the probe and the flight tip. Since the screw was double-flighted, during each rotation, we saw twice flights passing in front of the probe. Similar signals were obtained at Probes II and III locations. We used the first echo for dispersion monitoring, and used the second echo only for determining the clearance between the probe (or the barrel) and the flight tip. This distance, d , is given by

$$d = v \times \tau / 2, \tag{1}$$

where v is the velocity of ultrasonic waves propagating in the polymer and t is the time delay between the first and second echoes (which is identical to the round-trip ultrasound travel time between the probe and the flight tip). In Fig. 3, $\tau = 1.61 \mu s$. The ultrasound velocity in the polymer was determined at the extruder exit using the ultrasonic probes mounted on the instrumented die.

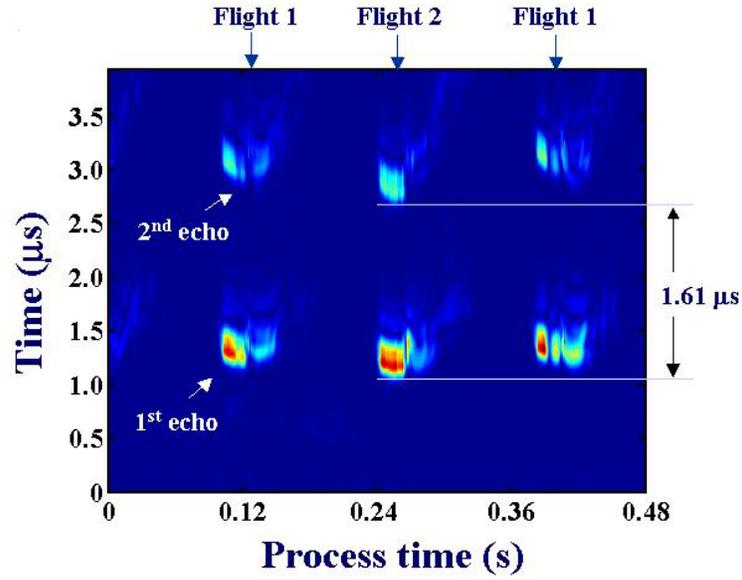


Figure 3. Evolution of echoes reflected from the tips of the screw flights at Probe I location.

In this work, we used the attenuation of ultrasonic waves in the polymer blends to determine the quality of dispersion. For each feed rate, we used the strength of the ultrasonic signals acquired during the extrusion of the pure HDPE as a benchmark for the evaluation of the strength of the ultrasonic signals acquired at the same probe location and during the extrusion of polymer blends at the same feed rate. More precisely, we used the relative attenuation of ultrasonic signal defined below:

$$\alpha = 20 \log(A_{pure} / A_{blend}) / (2d) , \quad (2)$$

where A_{pure} is the average of peak-to-peak amplitudes of all the echo signals acquired during the extrusion of the pure HDPE, and A_{blend} that of all the echo signals acquired at the same probe location and during the extrusion of a polymer blend at the same feed rate. Eq. (2) represents extra attenuation induced by the presence of a minor phase polymer in the polymer matrix in comparison with the case where no minor phase polymer is present.

Figures 4(a) and (b) show the variations of relative attenuations defined in Eq. (2) under two feed rates and four PS concentration levels.

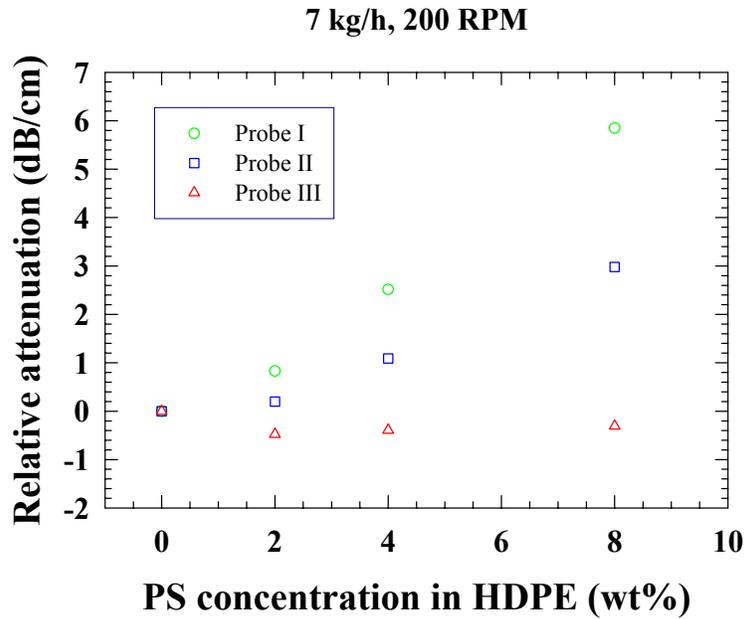


Fig. 4(a)

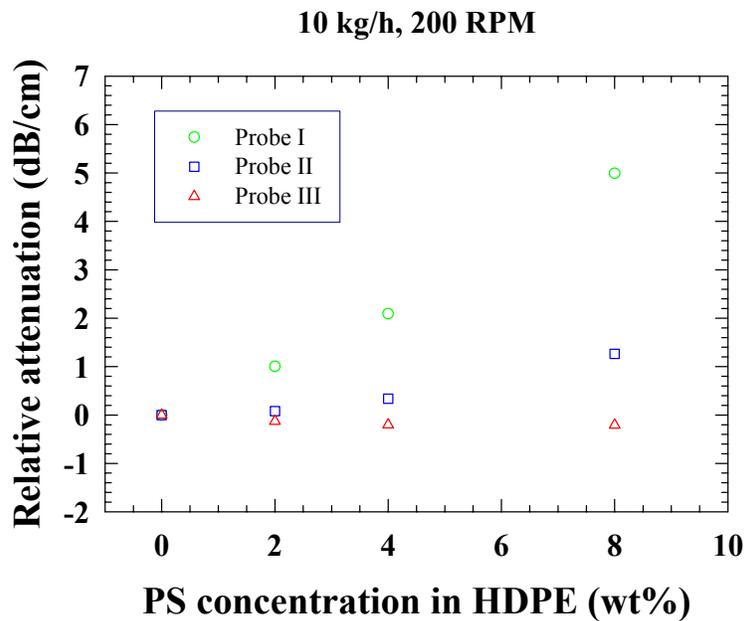


Fig. 4(b)

Figure 4. Variations of relative attenuations as a function of PS concentration at feed rates of (a) 7 kg/h, and (b) 10 kg/h.

As can be observed from Figures 4(a) and 4(b), at a given PS concentration, the relative attenuation at Probe I is always the largest in comparison with those at Probes II and III locations, whereas that at Probe III is always the smallest. Given that the dispersion at Probe I is the worst and that at Probe III is the best among the three locations, this means that that under the investigated conditions, the relative ultrasonic attenuation decreases with the improvement of blending quality. These results suggest that the relative ultrasonic attenuation can be used to monitor the progress of polymer blending along the melt flow channel inside the extruder. It is also interesting to

notice that at Probes I and II locations, the relative attenuation increases with the increase of PS concentration in HDPE. This could be quite understandable if one takes into account that, in molten state, PS has larger ultrasonic attenuation than HDPE and more PS in HDPE would simply mean higher ultrasonic attenuation. Also increasing PS concentration will create more boundaries between the minor and major phases, resulting in larger scattering loss of ultrasonic energy. However, the above observation does not hold at Probe III location where the relative attenuation seems to be insensitive to the PS concentration and even decreases slightly with the addition of PS. More studies will be needed in order to understand fully this phenomenon.

Figure 5 shows SEM pictures of some samples collected at the same cross-sections of the probes locations during extrusion of (a) 8% PS in HDPE at 10 kg/h feed rate and at Probe I location; (b) 2% PS in HDPE at 10 kg/h feed rate and at Probe I location; and (c) 2% PS in HDPE at 10 kg/h feed rate and at Probe III location. The small black dots in the pictures are the holes left by PS after being dissolved following 4 hour-immersion in cyclohexane at room temperature and thus represent the sizes and locations of PS particles. Much more PS particles are observed in Fig. 5(a) than those in 5(b) and 5(c) due to a higher PS concentration. Overall,

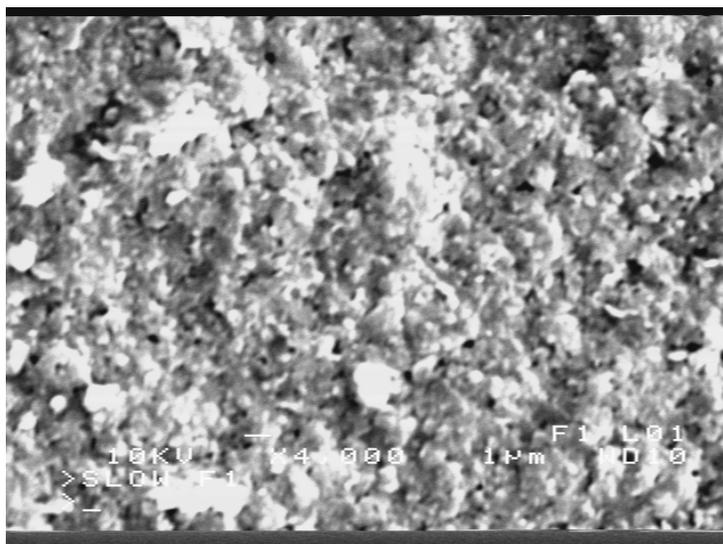


Fig. 5(a)

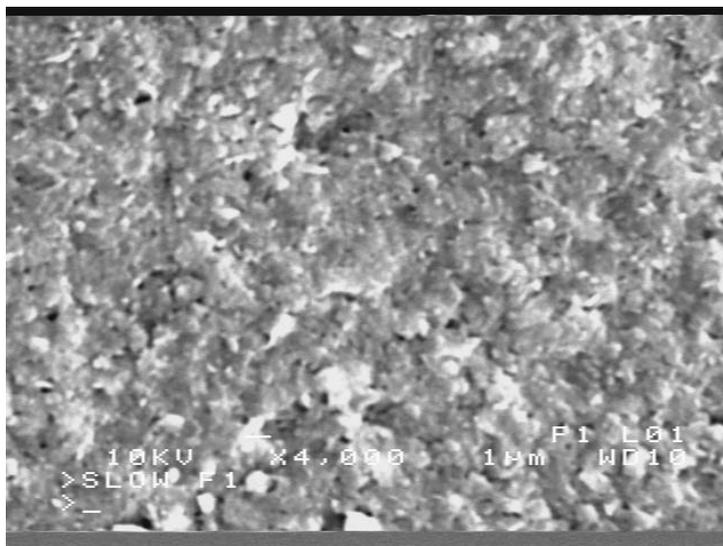


Fig. 5(b)

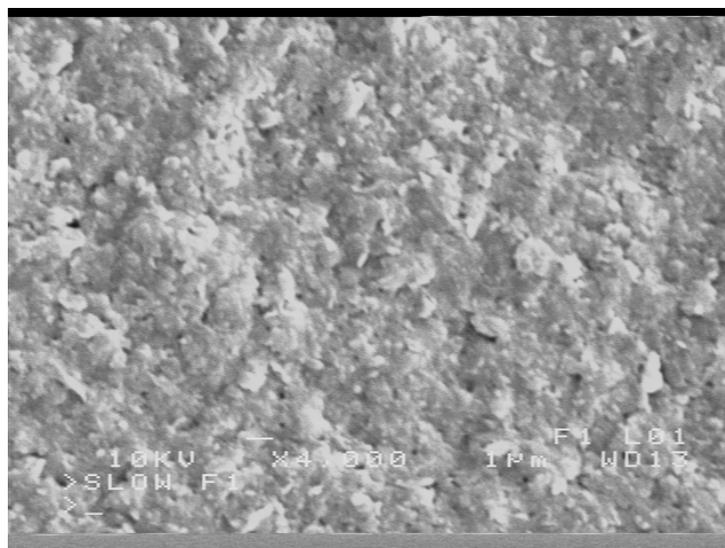


Fig. 5(c)

Figure 5. SEM pictures of some samples collected at the same cross-sections of the probes locations during extrusion of (a) 8% PS in HDPE at 10 kg/h feed rate and at Probe I location; (b) 2% PS in HDPE at 10 kg/h feed rate and at Probe I location; and (c) 2% PS in HDPE at 10 kg/h feed rate and at Probe III location.

the observable PS particles in Fig. 5(c) appear to be smaller than those in Fig. 5(b), owing to a better dispersion of PS in HDPE at Probe III location than at Probe I location. The difference between Figs. 5(b) and 5(c) can be seen clearly by ultrasound (Fig. 4(b)).

Conclusions: Ultrasonic monitoring of the blending process of a high-density polyethylene and a polystyrene has been performed at three locations along the polymer flow direction inside the barrel and at the exit of the extruder. The experiment results have shown that ultrasound can provide in-process information about the quality of dispersion of the minor phase polymer into the major phase polymer. Further studies are needed to explore fully the blending process related information provided by the ultrasonic technology presented in this paper.

Acknowledgments: The authors would like to thank Mr. Yuanbei Zhang for SEM analysis of samples. The financial support of Natural Sciences and Engineering Research Council of Canada is appreciated.

References: [1] J. Tatibouët and M. Huneault, “In-line ultrasonic monitoring of filler dispersion during extrusion”, Proc. Polymer Processing Soc. Conf., Montreal, Canada, May 2001..

[2] Z. Sun, C.-K. Jen, and C.-K. Shih, “Application of ultrasound in the determination of fundamental polymer extrusion performance: monitoring of melting and mixing processes”, Proc. Polymer Processing Soc. Conf., Guimariaes, Portugal, June 16-20, 2002.

[3] Z. Sun, J. Tatibouët, C.-K. Jen, “Application of ultrasound in the determination of fundamental polymer extrusion performance: monitoring of single-screw compounding extrusion”, Proc. Polymer Processing Soc. Conf., Guimariaes, Portugal, June 16-20, 2002.

[4] R.Gendron, J. Tatibouët, J. Guevremont, M.M.Dumoulin and L. Piché, “Ultrasonic behaviour of polymer blends”, Polym. Eng. Sci., vol.35, n°1, pp.79-91, 1995.