Ultrasound as a complementary tool to internal mixers for investigation of thermal mechanical degradation of PET

Sun, Zhigang; Zhao, Lijuan; Tatibouet, Jacques; Jen, Cheng-Kuei

http://dx.doi.org/10.1063/1.2964521

Ultrasound as a Complementary Tool to Internal Mixers for Investigation of Thermal Mechanical Degradation of PET

Zhigang Sun[a*], Lijuan Zhao[a,b], Jacques Tatibouët[a], and Cheng-kuei Jen[a]

[a] *Industrial Materials Institute, National Research Council Canada, Boucherville, Quebec J4B 6Y4, Canada*
[b] *The State Key Laboratory of Polymer Materials Engineering, Polymer Research Institute of Sichuan University, Chengdu 610065, China*

Abstract. An ultrasonically instrumented internal mixer was used to study thermal mechanical degradation of PET samples with different levels of water content and under different processing temperatures and blade speeds. The strength of ultrasound signals reflected from a roller blade of an internal mixer appeared to be more sensitive to PET degradation than the torque measurement means available on the internal mixer, suggesting that ultrasound could provide additional information on material property changes. A main advantage of ultrasonic degradation monitoring over torque measurement is that it could be implemented at various locations of an extruder to obtain localized melt degradation information.

Keywords: ultrasound, PET degradation.

INTRODUCTION

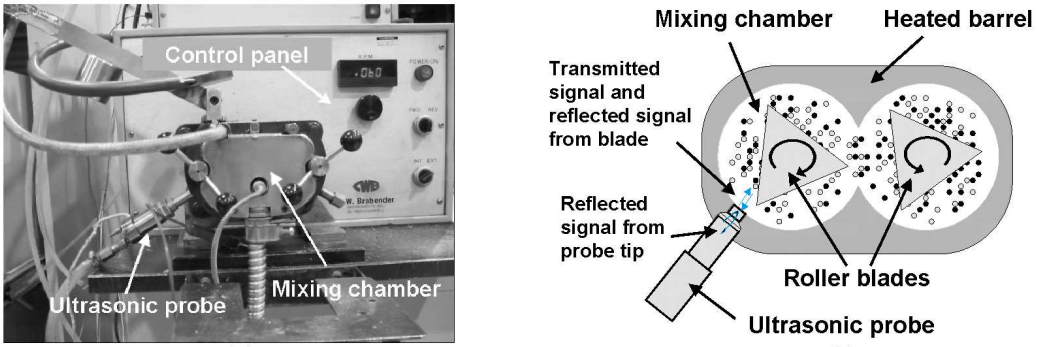
Internal mixer has been widely used in the plastics industry to study the melting, mixing, chemical reaction, and degradation of polymers through torque measurement. In the last couple of years, there are have been efforts to integrate an ultrasonic measurement system to a traditional internal mixer, with a view to provide complementary information which cannot be obtained with torque measurement. For example, a previous study showed that ultrasound was able to reveal information about a melting process taking place in the mixing chamber of an internal mixer that was not detectable by torque measurement [1]. It has also been reported that the state of dispersion of a mineral filler in a polymer could be much better sensed by ultrasound than by torque measurement. [2]. In this work, an ultrasonic system was used jointly with torque measurement for monitoring thermal mechanical degradation of PET samples under various water content and processing conditions of an internal mixer. The purpose was to see whether PET degradation was ultrasonically detectable and how sensitive was the ultrasonic approach compared to torque measurement.

EXPERIMENTAL SETUP AND MATERIALS

An ultrasonically instrumented internal mixer was used. Figure 1(a) and (b) illustrates respectively the external and internal views of the mixing chamber of the mixer. The ultrasonic probe sent ultrasonic pulse signals to the polymer and received echo signals reflected back either from the probe/polymer interface or from a rotating roller blade (Fig. 1(b)). The ultrasonic pulse signals were emitted at a rate of 720 pulses per rotation of the blade, resulting in an angular resolution of 0.5°. This resolution allows us to accurately track the echoes reflected from a specific region of the mixing blade (for example, the flight tip or root of the blade) and use the time of flight and amplitude of these echoes for process monitoring. The materials were dried and un-dried pellets of a same PET. When drying the sample, the sample was heated in vacuum at 60 °C for over 12 hours.

* Correspondence to: zhigang.sun@imi.cnrc-nrc.gc.ca

CP1027, *The XV[th] International Congress on Rheology, The Society of Rheology 80[th] Annual Meeting*
edited by A. Co, L. G. Leal, R. H. Colby and A. J. Giacomin
2008 American Institute of Physics 978-0-7354-0549-3/08/$23.00



(a) (b)

FIGURE 1. External (a) and internal (b) views of an instrumented mixing chamber installed on an internal mixer.

RESULTS AND DISCUSSIONS

Figure 2 shows torque and ultrasound echo amplitude measurements results at a set chamber temperature of 260 °C and blade speed of 70 RPM, for a dried and an un-dried PET samples, respectively. The relative echo amplitude used in the figure, as in the remaining figures, is defined as the ratio of the amplitude of an ultrasonic echo signal reflected from a blade flight tip to that reflected from the ultrasonic probe/polymer interface. The ultrasonic signal strength was little or not detectable during the initial stage of the heating. After 4 minutes into the heating, the signal strength increased sharply, suggesting that the PET sample was getting melted more and more uniformly, given that the more uniform the polymer melt is, the less acoustic energy loss at the boundaries of unmelts. At about 5.5 minutes into the heating, the un-dried sample reached a plateau (indicated with arrow 1), suggesting that the PET sample was melted completely. The dried PET sample reached a plateau about 1 minute later, as indicated by arrow 2, suggesting that it took longer for the dried sample to melt completely. For the un-dried sample, the plateau continued till about 8.5 minutes into the heating, then the signal strength started to decrease. The transition point, indicated with arrow 3, could be the starting point of degradation of the un-dried PET sample. For the dried PET sample, this transition point, indicated with arrow 4, appeared significantly later, at about 11 minutes into the heating. This is in accordance with a widely accepted understanding that a dried PET is less susceptible to thermal mechanical degradation that PET with a certain degree of water content. It is interesting to notice that this transition point (arrow 3) for the un-dried PET was not sensed by the torque measurement, whereas in the case of dried PET sample, the torque happened to show a transitional behavior at about the same time. In the figure, the ultrasonic signal strength fluctuates noticeably. This fluctuation is believed to be caused by the vibration of the roller blade during rotation. Figure 3 displays the results for dried PET samples at 70 RPM for two different temperatures. As can be seen in the figure, at a higher set temperature of mixing chamber (270 °C), the ultrasound signal strength reached a plateau (arrow 1) earlier than at a lower temperature (260 °C, arrow 2), indicating that the sample melted more quickly at a higher temperature. Also the sample reached the transition point faster at a higher temperature (arrow 3 compared to 4). Figure 4 shows the results for un-dried PET samples at 70 RPM, and at 260 and 270 °C, respectively. In the case of 270 °C, the polymer appeared to degrade so quickly that the melt even didn't have time to stabilize (arrow 1 and 3 together). When lowering down the temperature to 260 °C, the polymer appeared to melt completely at about 5.5 minutes into the heating (arrow 2) and enter the degradation stage at about 8.5 minutes (arrow 4). Again, the transition points seen by ultrasound were not detectable by torque measurement. Figure 5 displays the results for un-dried PET samples at 270 °C, and at blade rotation speeds of 40 and 70 RPM, respectively. At 270 °C, the degradation appeared to happen at the same time the sample got completely melted, and this for both blade speeds. Although in the case of 40 RPM the sample appeared to reach complete melting later than in the case of 70 RPM, the results seem to suggest that once the sample reached complete melting, the melt degraded faster in the case of 40 RPM than in the other case.

CONCLUDING REMARKS

The ultrasonic approach investigated in this work appeared to be able to sense the melting and degradation status of PET samples in a way that torque measurement could not. Further evidence relating ultrasonic signature to molecular weight or morphology of PET samples collected at different stages into the heating is needed to confirm

the applicability of ultrasound for PET degradation monitoring. A potential beneficial characteristics of an ultrasonic technology is that it can be implemented easily at different locations of an extruder to capture localized melt quality information.

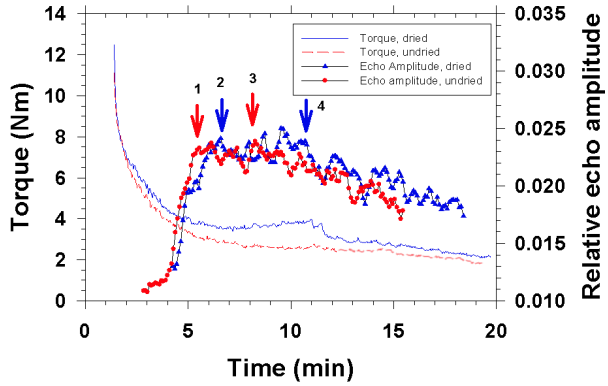


FIGURE 2. Torque and ultrasonic echo amplitude measurements results at 260 ℃ and 70 RPM for dried and un-dried PET samples.

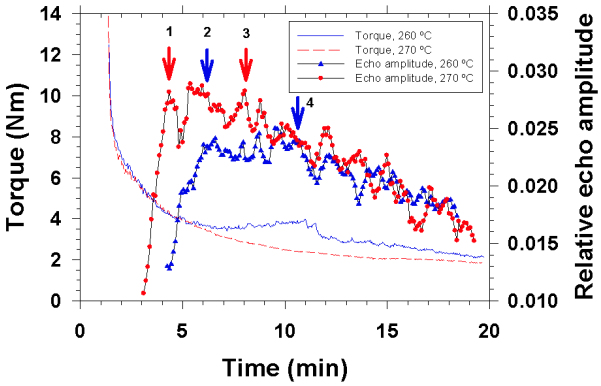


FIGURE 3. Torque and ultrasonic echo amplitude measurements results for dried PET samples at 70 RPM for two different temperatures.

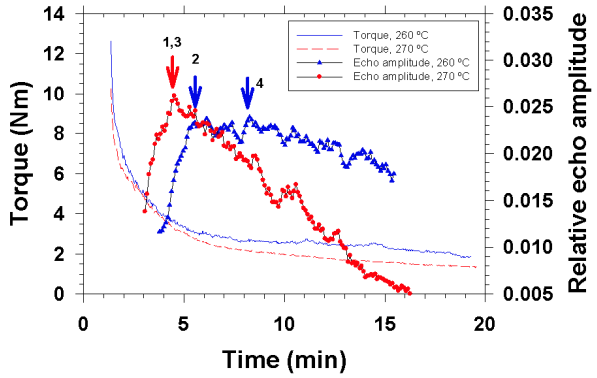


FIGURE 4. Torque and ultrasonic echo amplitude measurements results for un-dried PET samples at 70 RPM and two different temperatures.

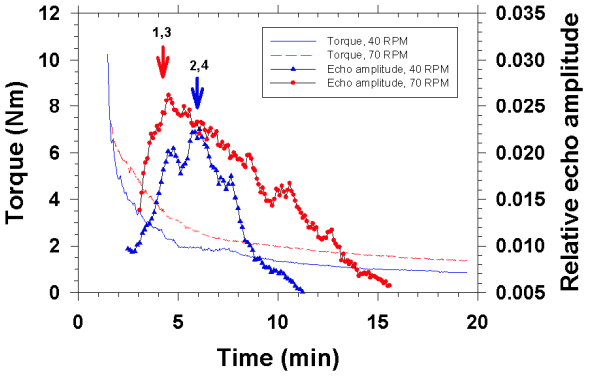


FIGURE 5. Torque and ultrasonic echo amplitude measurements results for un-dried PET samples at 270 ℃ and two different blade rotation speeds.

ACKNOWLEDGMENT

Lijuan Zhao acknowledges the financial support from Chinese Scholarship Council (CSC).

REFERENCES

1. C.-K. Shih, J. Tatibouët, Z. Sun, and C.-K. Jen, *Proc. Polymer Processing Soc. Conf.*, Guimarariaes, Portugal, paper#142, pp.1-7, June 16-20, 2002.
2. Z. Sun, C.-K. Jen, H. L. Liang, and C.-Y. Su, *Proc. Int' Conf. on Dynamics, Instrumentation and Control*, Nanjing, China, paper#FE01-2, pp.1-7, Aug. 2004.