

A Novel Approach for Observing Morphology of Polypropylene under Ultrasonic Vibration by SALS

Li Fang TONG¹, Ye SHEN², Qiang ZHENG^{1*}, Yong Gang SHANGGUAN¹

¹Department of Polymer Science and Engineering, Zhejiang University, Hangzhou 310027

²First Affiliated Hospital, Zhejiang University College of Medicine, Hangzhou 310027

Abstract: In this paper, a set of ultrasonic vibration device connecting with small angle light scattering(SALS) for *in-situ* observing phase-transition of polymer under ultrasonic vibration was established. The experiments for verifying polypropylene(PP) indicated that ultrasonic vibration resulted in the decrease of the crystallization rate and made PP crystallizable at low temperature.

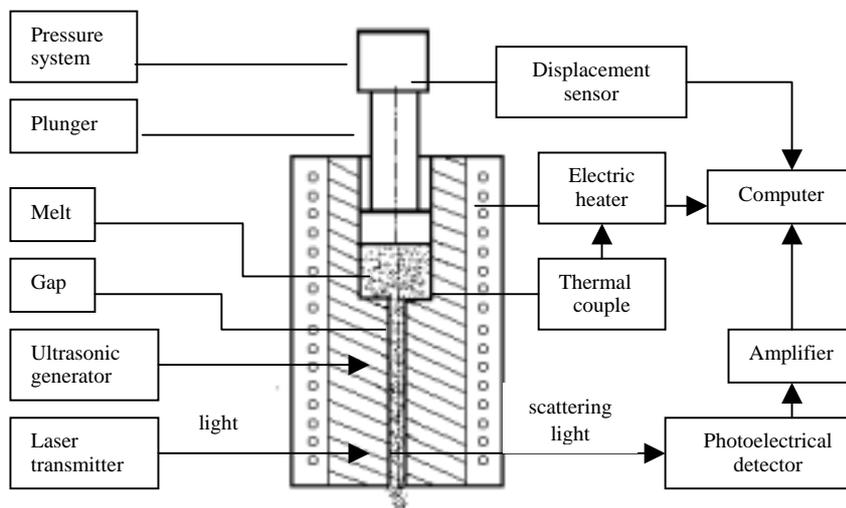
Keywords: Polypropylene, ultrasonic, vibration, crystallization, SALS.

Recently, there have been extensive interests in the application of ultrasonic to academic and applied research of polymers concerning depolymerization, synthesis, measurement, etc¹⁻⁴. Isayev *et al.*⁵ introduced ultrasonic vibrations to an extruder, and found that the *in-situ* copolymer formed at the interface of an immiscible polymer system consisting of PP/natural rubber could improved the interfacial adhesion, morphology and mechanical properties of the system. In this paper, we propose a novel approach to *in-situ* observation of morphology for PP using a set of ultrasonic vibration device connecting with small angle light scattering (SALS) established by our research group and discuss primarily the effect of ultrasonic vibration on non-isothermal crystallization for PP.

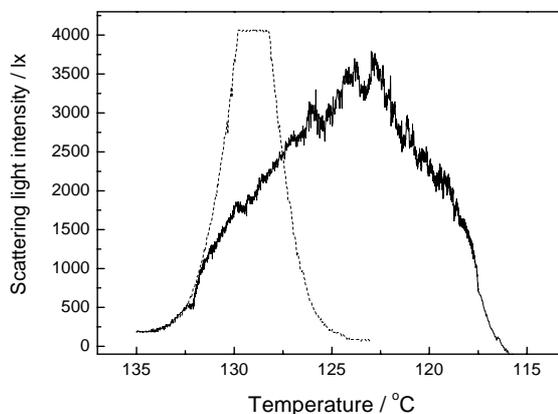
The ultrasonic vibration device was illustrated in **Figure 1**. The device consisted of a gap extruder, an ultrasonic generator and a SALS system. The dimension of the gap was 0.8 (h) × 12 (w) × 60 (l), w / h = 15 > 10, and could be considered as a boundless parallel plates system. The signals of displacement sensor, electric heater and photoelectrical detector were sent to a computer for data record and processing. The ultrasonic frequency and power range were 20 kHz and 0 ~ 100 W, respectively. The direction of ultrasonic vibration was controlled vertically to that of melts flow.

The PP samples used in the test for verifying the validity of the device were commercially available products supplied by Sinopec Shanghai Petrochemical Company Limited (PPH-T-022--A). PP melts were extruded to fill up the gap at 200°C. For simplifying the effect factors of experiments, the PP melts was relaxed for 20 min at 200 °C to eliminate the effect of pressure, then the temperature was decreased at 2°C/min. The scattering light arisen from the PP melts was detected by SALS. When PP melts

* E-mail: zhengqiang@cmsce.zju.edu.cn

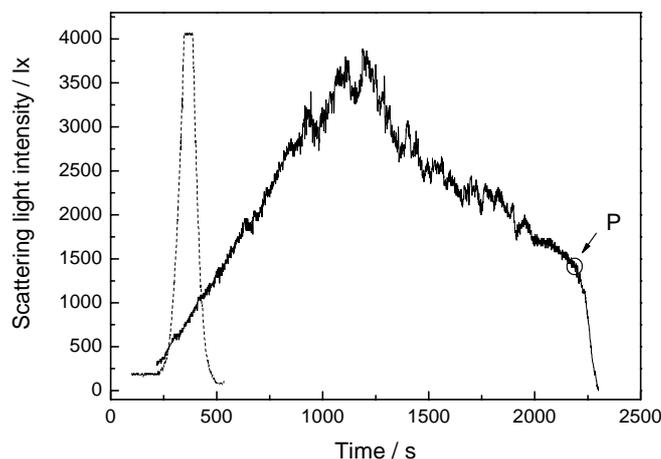
Figure 1 The illustration of the ultrasonic vibration device.

began to crystallize at crystallization temperature, the density of the PP melts fluctuated, which induced the change of the scattering light intensity. With the increase of spherulitic size and number, the scattering light intensity increased first and decreased afterwards since the multiple scattering occurred. In order to detect the effect of ultrasonic vibration on crystallization of PP, the ultrasonic vibration was added to the system when the scattering light intensity increased, namely, the crystallization of PP began under the ultrasonic power of 100 W. Simultaneously, the sample not undergoing the ultrasonic vibration was prepared at the same condition for comparison. **Figure 2** shows the relationship between temperature and the scattering light intensity for PP under non-isothermal crystallization with and without ultrasonic vibration. The results reveal

Figure 2 The relationship between temperature and the scattering light intensity for PP with non-isothermal crystallization under different conditions: no ultrasonic vibration (dash line); ultrasonic vibration (real line)

that the crystallization temperature of PP under ultrasonic vibration increases as compared with that without vibration. On the other hand, the crystallization for the samples without vibration finished at 124°C, while for the samples vibrated under ultrasonic, finished at 115.5°C, indicating ultrasonic could result in PP crystallizable at relatively low temperature because of the high energy of ultrasonic. **Figure 3** gives the time dependence of the scattering light intensity for PP under non-isothermal crystallization with and without ultrasonic vibration. It is clear that the increase of the scattering light intensity with time under ultrasonic vibration is more slowly than that without vibration. The phenomena above imply that the crystallization rate of the samples decrease, and crystallization of PP needs more time under ultrasonic vibration, as compared with those without ultrasonic vibration. Combining **Figure 2** it is believed that ultrasonic make the cooling rate descend from 2°C/min to 0.5°C/min. The reason for this is because ultrasonic vibration makes the temperature of melts increase; in other words, this is the reason why crystallization rate becomes slow under ultrasonic vibration. However, when the ultrasonic vibration is ceased at point P as marked in **Figure 3**, the scattering light intensity decreases sharply within a few seconds. Hence, we conclude that the decrease of the scattering light intensity is not only resulted from the decrease of temperature, but also from the disappearance of ultrasonic vibration during the period of PP crystallization, since the temperature could not decrease sharply in such a short period.

Figure 3 The dependence of the scattering light intensity on time for PP with non-isothermal crystallization under different conditions: no ultrasonic vibration (dash line); ultrasonic vibration (real line)



Acknowledgments

This work was supported by the National Science Funds for Distinguished Young Scholars (grant 50125312), Key Program of National Natural Science Foundation of China (grant 50133020) and the Special Funds for Major State Basic Research Project, China (grant G1999064800).

References

1. A. Grönroos, *et al.*, *Ultrasonics Sonochemistry*, **2001**, 8, 259.
2. G. J. Price, *Ultrasonics Sonochemistry*, **1996**, 3, S229.
3. C. Bockenheimer, *et al.*, *International Journal of Adhesion & Adhesives*, **2002**, 22, 227.
4. T. Kobayashi, X. Chai, N. Fujii, *Separation and Purification Technology*, **1999**, 17, 31.
5. A. I. Isayev, Jeong Seok Oh, M. A. Rogunova, *Polymer*, **2003**, 44, 2337.

Received 16 June, 2003