Ultrasound-Enhanced Devolatilization of Polymer Melt

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Introduction

Polymer manufacturing processes often include a devolatilization (DV) stage for removing low molecular weight substances from the polymer melt. As a general rule, the system subjected to a DV process is a polymer melt containing dissolved impurities in quantities not exceeding fractions of one percent. In practice, DV is carried out in special equipment, by exposing the polymer melt to vacuum (Biesenberger and Sebastian, 1983), leading to a foaming-boiling DV mechanism (Lee and Biesenberger, 1989). To bring about foaming, the temperature is raised (in the permissible range) and the ambient pressure is decreased, creating supersaturation conditions. The lower the initial volatile concentration in the polymer, the higher the vacuum required. It appears that the equilibrium gaseous nuclei within the polymer melt are activated into bubble growth centers when the melt is superheated. When pressure is decreased to zero, and, furthermore, with transition to negative pressures (volume stretching of the material), smaller and smaller nuclei may become activated, sharply increasing the number of bubble growth centers. As a result, DV rate should also increase, especially when volatile concentrations are low and nucleation rate is the determining factor.

Volume stretching of a liquid can be practically achieved by means of a powerful ultrasound source. The acoustic field causes high-frequency stretch-compression stresses within the liquid; the stresses may upset the continuity of the liquid, and as a result, areas of tiny bubbles arise in the liquid (acoustic cavitation). The phenomenon, thoroughly investigated for low-viscosity liquids (Knapp et al., 1970), can also be observed in polymer melts (Peshkovskii et al., 1983). The idea of intensifying foaming by ultrasonic acoustic field during polymer melt DV, is the subject of this article.

Experimental Setup and Procedure

The experimental setup shown schematically in Figure 1, was designed to extrude an acoustically treated (or untreated) polymer melt into a vacuum chamber. The 20 mm diameter single screw extruder was equipped with a cross head die incorporating a waveguide attached to an ultrasound generator and acoustic transformer. The waveguide was a longitudinally oscillating 10 mm diameter titanium rod immersed in the polymer melt. The vacuum chamber attached to the cross head was removable, and it was equipped with a special sample holder.

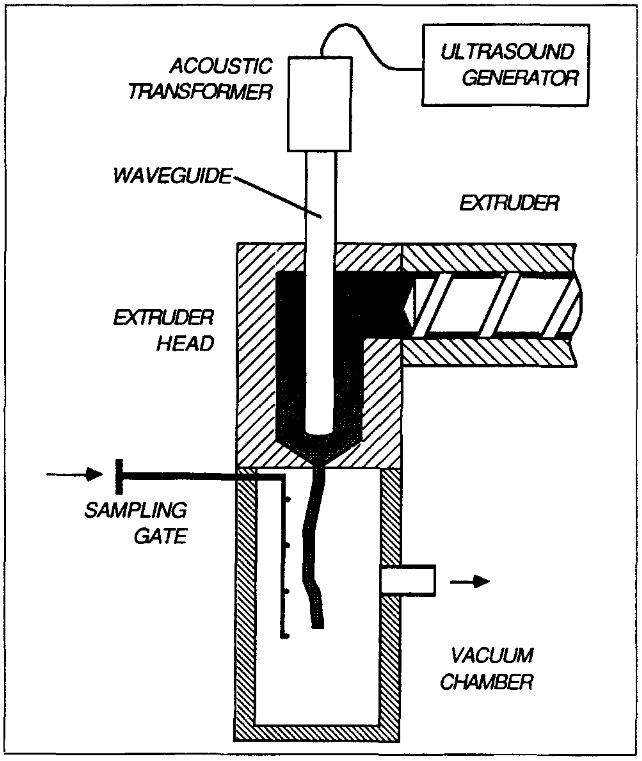

Figure 1. The experimental installation.

Electric heaters controlled the temperatures of the cross head and the vacuum chamber. In experiments without ultrasonic treatment, the temperature of the head was set higher, to compensate for the temperature increase of the polymer melt as a result of ultrasonic treatment. Thus, the temperature of the extruded strand, measured by a needle thermocouple, was the same for ultrasonically treated and untreated melts. In the experiments reported in this note, temperature was 255°C and ultrasound frequency 20 kHz. The material used was com-

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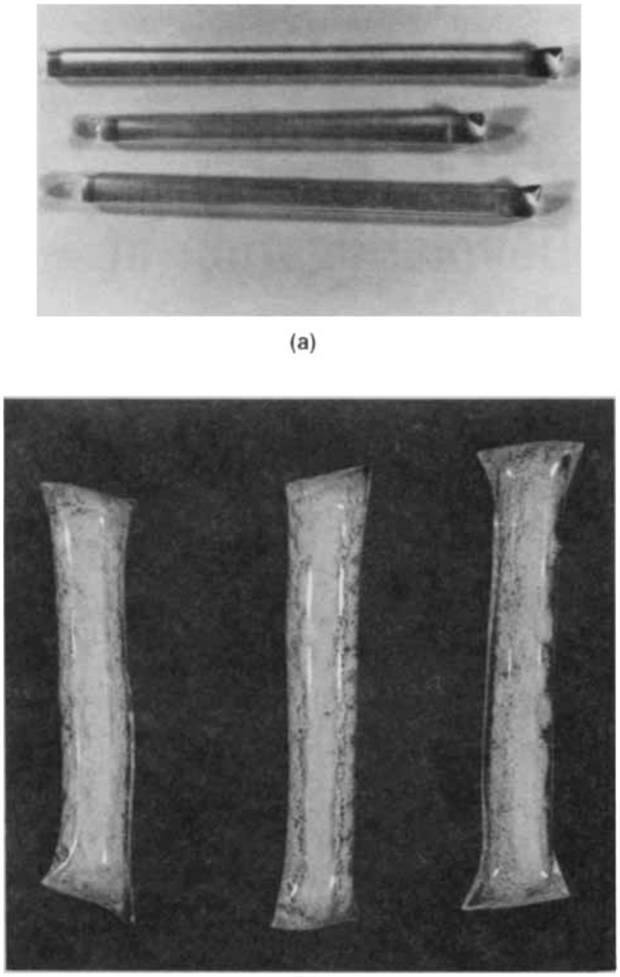
(a)

(b)

Figure 2. Polystyrene samples extruded at the same melt temperature 255°C, without vacuum, in the absence (a) and in the presence (b) of ultrasound.

mercial polystyrene (HH102E produced by the Israeli Petrochemical Enterprises) with a residual styrene content of 750 ppm and enriched with styrene up to 3,000 ppm. The latter was prepared by storing the pellets in a closed vessel saturated with styrene vapors (Albalak et al., 1990).

Experimental Results and Conclusions

A series of experiments was carried out to study the effect of the acoustic treatment on the devolatilization process.

First melt strands were extruded into the open with and without the acoustic treatment. Figures 2a and 2b show the samples obtained. Clearly, the acoustically treated samples visibly foam up even at atmospheric pressure conditions; whereas, their untreated counterparts show no sign of foaming. These experiments, therefore, show a qualitative difference, in devolatilization of acoustically treated and untreated polymer melts.

In order to study the quantitative effect of the acoustic treatment, the strands were extruded into the vacuum chamber and exposed to 150 mm Hg, 50 mm Hg, and 12 mm Hg vacuum levels, for periods of time from 5s to 40s. Subsequent to reaching a steady state, the extruder was switched off and the melt strand sample was taken by a specially designed "ladder-like" sample holder. This was attached to a rod which when inserted shut off the die exit and spliced off the melt strand sample. The samples, dissolved in dichloromethane, were then analyzed for residual styrene by gas chromatography.

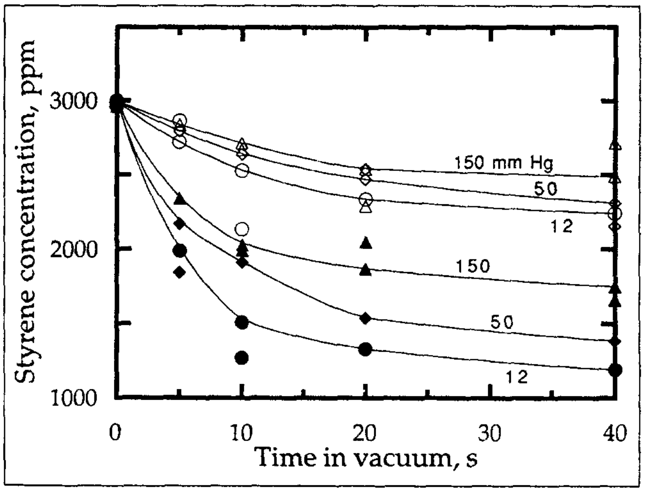

Figure 3. Residual styrene concentration in PS extruded at 255°C.

The open symbols refer to experiments without ultrasound; the filled ones - to experiments with ultrasound treatment. The parameter is the absolute pressure in the vacuum chamber (mm Hg).

The results shown in Figure 3 indicate that acoustic treatment significantly reduces the level of the residual styrene and that the effect is amplified with increasing vacuum levels. Considering the timescale at which the phenomenon occurs, it can be concluded that acoustic treatment may be an important and practical means for improving polymer melt strand devolatilization.

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