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Ultrasonic Measurements During Solidification of Molten Polyethylene Using Polymer Waveguide Probe

ポリマー導波プローブを用いた溶融ポリエチレンの凝固中の超音波測定

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1. Introduction

Polymers such as polyethylene, polystyrene and polyurethane, because of their excellent moldability and inexpensiveness, have widely been used as an indispensable material for various industrial products such as aircrafts, automobiles as well as daily necessities. In general, the mechanical properties of polymers strongly depend on the orientation of polymer chain that is also related to its manufacturing process. Therefore, there are increasing demands for measuring and characterizing the properties and conditions of polymer resin during solidification process, to assure the quality and reliability of polymer products. In particular, a noninvasive technique for monitoring such molten polymer is greatly desired for some practical application in industries as well as academic fields. However, there have been little technique to meet such requirements.

Ultrasound is considered to be a potencial candidate for such monitoring of molten polymer because of its capability to probe the interior of materials and its high sensitivity to material properties. The advantages of using ultrasonic measurement method, such as non-invasiveness, faster time response and safety are also quite attractive for engineering applications. In our previous works, we have developed ultrasonic buffer rod as an effective ultrasonic measurement means for high temperature materials to which conventional ultrasonic transducers cannot be easily applied [1–8]. Such ultrasonic buffer rods were also successfully applied to several materials and process monitoring at high temperatures. In this work, a polygonal buffer rod has been applied to molten polymer to measure the variations in the ultrasonic velocity and attenuation of the solid and liquid phases. In addition the behaviour of the solid/liquid interface during solidification process of molten polymer is examined.

2. Experiment

Ultrasonic pulse-echo measurements are applied to molten polymer monitoring during cooling process. **Figure 1** shows the schematic diagram of experimental set up for the ultrasonic measurement of molten polymer resin. A low density polyethylene (LDPE) is employed as a polymer specimen. The polymer is first melted in a container whose bottom is heated by an electric heater and then the same bottom is considerably cooled by water so that the molten polymer could gradually be solidified. It is expected that unidirectional solidification will occur owing to the bottom cooling. In the ultrasonic measurements, a heptagonal taper buffer rod [9, 10] made of polyimide that effectively reduces trailing echoes accompanying with the main pulse echo and provides appropriate measurements with high signal to noise S/N ratio, is employed. A 1 MHz piezoelectric transducer for longitudinal wave is installed on the top of the buffer rod. The buffer rod is immersed into the molten polymer and ultrasonic pulse echoes are continuously acquired every 2 ms with a PC-based real-time acquisition system. The sampling rate for the ultrasonic signal acquisition is 100 MHz. Spurious fluctuation in the ultrasonic signal due to electrical noise in the measurements is reduced by taking the average of 100 times signals. It is noted that the polyimide buffer rod provides a better acoustic impedance matching between the rod and LDPE. Five sheath thermocouples are inserted into the upper and bottom portions of the LDPE to measure the temperature distribution in the ultrasonic propagation region.

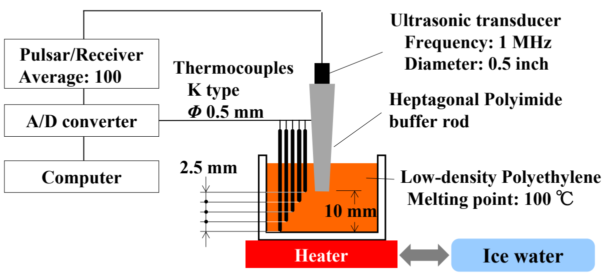

Fig. 1 Schematic diagram of the experimental set up used for molten polymer measurements.

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3. Results

Figure 2 shows ultrasonic pulse echoes measured before and after the immersion of the tip of the buffer rod into the molten LDPE at temperature around 470 K. When the rod tip is immersed into the molten LDPE, a series of clear echoes from the bottom of the container can be successfully obtained. The cross-correlation method is applied to the two bottom echoes, A_1 and A_2, to determine the longitudinal wave velocity of the LDPE. The attenuation coefficient of the LDPE is also determined from the bottom echoes. **Figure 3** shows the temperature dependences of the velocity and attenuation coefficient of the LDPE. It has been found that the velocity in solid state decreases significantly with temperature rise, whereas it decreases slightly in liquid state. The attenuation also shows a strong temperature dependence.

Figure 4 shows the variation in the measured pulse echoes with elapsed time when the bottom surface of the molten LDPE is being cooled by water. It can be seen that a small echo from a solid-liquid interface is observed at each time step and such echo moves to the left-hand side because of the growing of solidification interface. Thus, it has been demonstrated that the present ultrasonic buffer rod could be a useful tool to monitor the molten polymer during heating and cooling processes.

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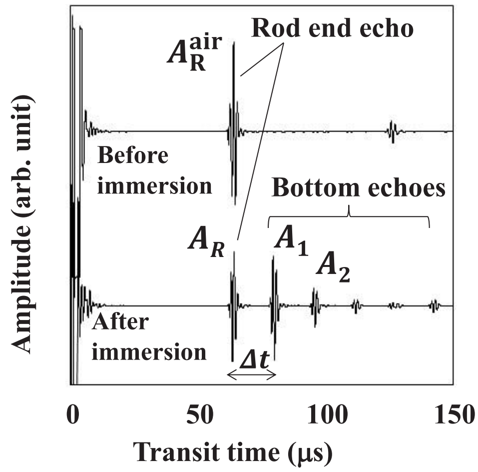

Fig. 2 Ultrasonic pulse echoes before immersion into molten LDPE (upper) and after immersion (lower).

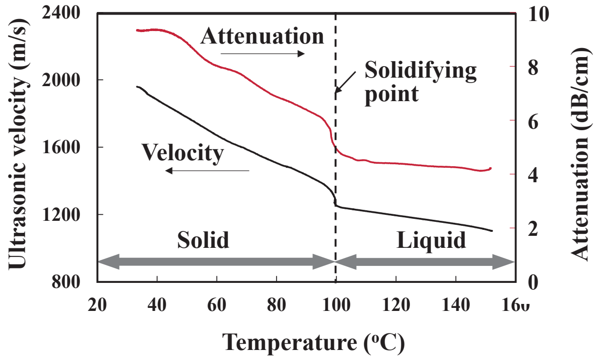

Fig. 3 Temperature dependences of the ultrasonic velocity and attenuation coefficient of the LDPE.

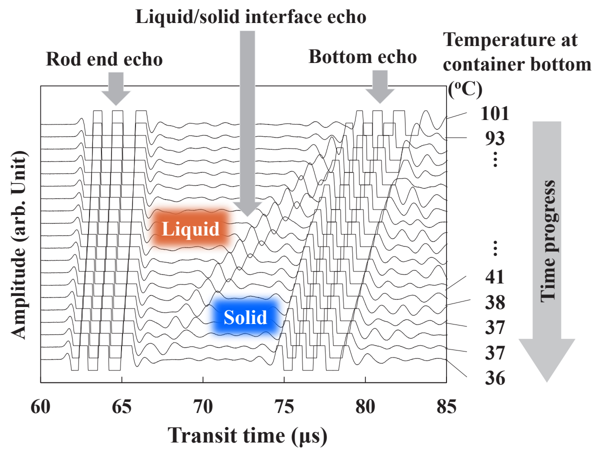

Fig. 4 Variation of the measured interface echo with elapsed time during unidirectional solidification process.