In-situ Observation of One-shot Chemical Foaming Process by X-ray Transmission Imaging

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Abstract. Visual observation experiments of the one-shot chemical foaming process were performed by the X-ray transmission imaging method. Specimen compound was composed by the metallocene linear -low -density poly-ethylene (LLDPE) containing the chemical foaming agent (CFA), the CFA activator and the cross-linker. Foaming was carried out in the sealed cavity with the X-ray detecting system and the pressure sensor. The visualized image changed from the homogeneous melt state to the white spot pattern. It was shown that the low electron density structure (i.e. bubble) was formed by the generated gas resulting from CFA thermal decomposition.

Keywords: chemical foaming, X-ray imaging, cell structure, polymer foam
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INTRODUCTION

Chemically cross-linked poly-olefin foams by the one-shot chemical foaming process have been widely used for shock attenuation parts of shoe soles. A typical one-shot chemical foaming process involves several steps. First, polymer compounds including the chemical foaming agent (CFA), the CFA activator and the polymer cross-linker are filled into the mold at a certain temperature. Second, cross-linking reaction of the polymer and thermal decomposition reaction of the CFA proceed simultaneously, while suppressing expansion of compounds by the mold clamping pressure. Finally, as the mold clamping pressure is released, the compounds immediately expand, and are cooled. Then cross-linked polymer foams are obtained. Concerning the one-shot chemical foaming process, the forming mechanism of the cell structure has not been revealed yet. In previous studies, foaming behaviors on one-shot chemical foaming process have been investigated by considering molding conditions and viscoelasticity of compounds [1, 2]. And we reported that thermal decomposition behaviors of the CFA during the one-shot chemical foaming process by the constructed in-situ measuring system [3]. In this study, visual observation of the one-shot chemical foaming process was performed by an X-ray transmission imaging method to investigate the cell forming mechanism on the one-shot chemical foaming process.

EXPERIMENTAL

In-situ observation experiments were performed at SPring-8 BL24XU (Hyogo Prefecture Beam Line, Proposal No. 2014A3329, 2015A3267 and 2015B3267). The visual observation system consists of a transmitted X-ray detector, and a cavity equipped with a pressure sensor and a temperature control unit. The cavity constructed by the stainless steel is cuboid shape with 5mm length, 5mm width and 1.5mm thick, and it has a pressure sensor (KISTLER, 6190CA). A part of the cavity is constructed by the glass to ensure X-ray transmittance. The transmitted X-ray is detected by the CMOS camera (HAMAMATSU PHOTONICS K.K., Orca-Flash4.0, 2048 × 2048pix, view size is 1.3 × 1.3mm) at 0.5s intervals. The X-ray energy is adjusted to 15keV. The cavity temperature was controlled at 433K. The specimen compound is based on a commercial metallocene linear -low -density poly-ethylene (LLDPE, MFR = 3.8g / 10min). Dicumyl peroxide (DCP, 1 min half-life temperature = 448.2K), azodicarbonamide (ADCA, median size = 6μm), and zinc oxide (ZnO) were used as the cross-linker, the CFA and the CFA activator, respectively. The specimen was blended by using open two roll mills at 393K on following weight ratio; LLDPE/DCP/ADCA/ZnO = 100/1.2/10/5. The specimen compound did not leak from the cavity during observation experiments.

RESULTS AND DISCUSSION

First, it was verified to reproduce the one-shot chemical foaming process in this measurement system. Fig. 1 shows the cavity pressure *P* profile at 433K. Arrows denote the timing corresponding to images of Fig. 2. *P* showed s-shaped behavior; after the induction phase, *P* increased rapidly, then, reached the saturated value. This *P* profile resulted from the CFA thermal decomposition behavior as described previous studies [3, 4]. The obtained polymer foam after process observation experiment had a closed cell structure. We confirmed that this one-shot chemical foaming process has reproducibility in our constructed observation system of this study.

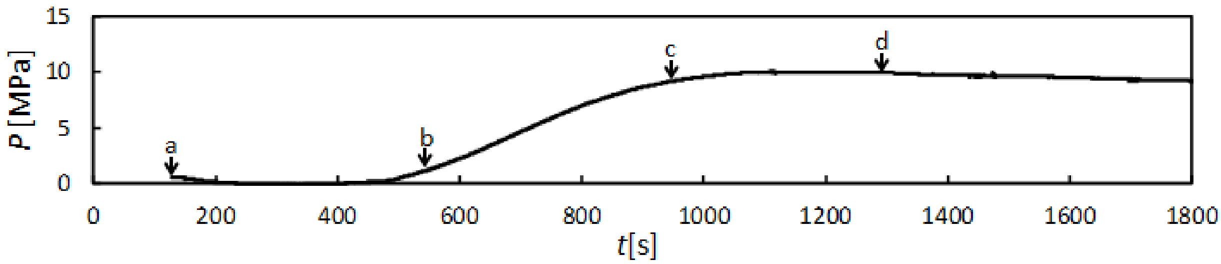

FIGURE 1 Cavity pressure profile of LLDPE compound at 433K. Arrows denote the timing corresponding to images of Fig. 2.

A series of cavity internal image at 433K is shown in Fig. 2. Fig. 2(a) at the induction phase shows a homogeneous image. The CFA particle was not able to be confirmed clearly in this observation system. Then, in Fig. 2(b), white spots with a diameter of tens microns appeared from lower right of the image. The appearance of the white spot pattern indicated that the low electron density structure was formed at this stage in the cavity. Considering *P* profile in Fig. 1, CFA thermal decomposition started at around 530s. We have concluded that the low electron density structure resulted from the generated gas by CFA thermal decomposition; the appearance of the white spots indicated the forming an initial cell structure in the cavity at this stage. AS can be seen in Fig. 1, from this stage the pressure steeply increased toward the pressure saturate phase. In Fig. 2(c), the number of white spots increased, and appeared all over the observation area despite of high pressure. Finally, in Fig. 2(d), there was little change in the white spot morphology with that of Fig. 2(c). The cell collapse and coalescence couldn't be observed in this observation level and we consider that the formed initial cell structure was maintained during foaming process in the high pressure cavity.

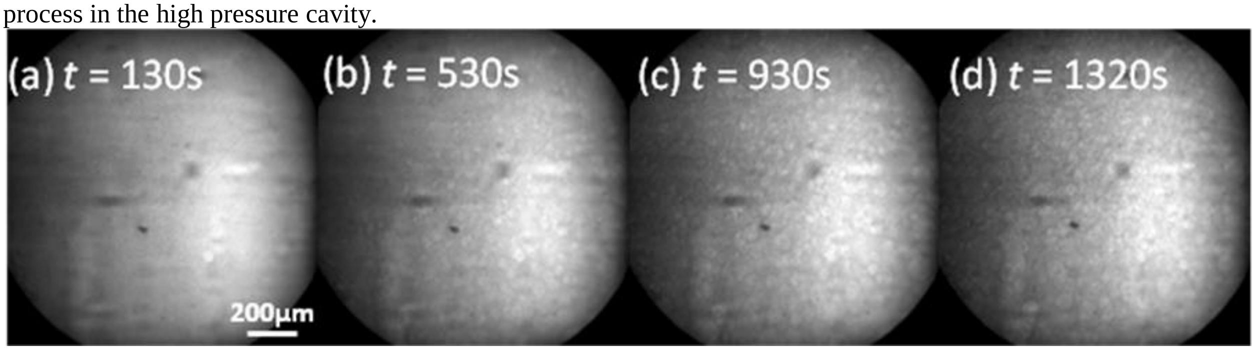

FIGURE 2 A series of X-ray transmission image of one-shot chemical foaming process at 433 K (a-d).

CONCLUSIONS

The one-shot chemical foaming process was investigated by the in-situ X-ray visual observation method. We showed that cells generated by the gas of CFA thermal decomposition were formed in the early low pressure stage and number of the cells increased in the pressuring stage in the cavity, while there was little change in the cell size.

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