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Experimental study of cure process of foam rubber: observation of cell structure

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Abstract

Experimental study has been made during cure process of SBR/NR foam rubber. Rubber sample with 30 mm thick, made up by stacking thin rubber sheets in layers, was packed in a metal mold, and peroxide cure was performed by transient heat conduction. Rubber heating time was changed in several steps in order to study the effects of the cure time on the blowing characteristics. Also swelling test was conducted in order to study the relation between the cell structure and the crosslink density. Typical temperature field of one-dimensional, transient heat conduction was observed, and results of the observation studies showed that the cell structure changed depending on both the position from the heating surface and the heating time, and two extreme cell structure, open-cell foam, and closed-cell foam, was clearly observed. Image analyses of the cell structure showed that the porosity distribution increased with the increase of the distance from the heating surface, and the porosity was lower for longer heating time at a same position. Average area of the foam almost took similar results for the result of the porosity, and these various quantities were correlated to the crosslink density.

Keywords: Foam rubber; Cell structure; Crosslink density; Image analysis

1. Introduction

Foam rubber is wide spread, ranging from household, sports to industrial regions. Processing of foam rubber has been considered to be more complex than that for solid rubbers because the processing includes foaming in addition to compounding and curing. This leads to developing empirical methods in foam rubber processing. Cell structure in form rubber is usually divided broadly into open-cell foam and closed-cell form, and qualitative findings have been provided that the former is predominant for low viscosity region and the latter for high viscosity region [1]. However, engineering-based technical information about foam rubber processing seems to be few. The present authors [2] have conducted experimental and numerical studies on the curing process of solid, styrene-butadiene rubber having relatively large size and have proposed a prediction method for the process.

In the present work, an experimental study on the foam rubber processing has been conducted. As a first step, observations of cell structure, measurement of temperature field, and evaluation of crosslink density were conducted simultaneously. Results of the observation studies are correlated to the crosslink density.

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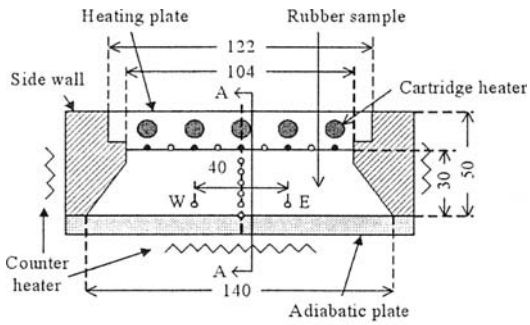
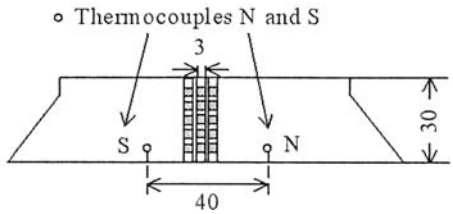
2. Experimental apparatus and procedure

Fig. 1 illustrates cross-section of the mold used for the experiments. The mold consisted of aluminum-alloy heating plate, side wall made of stainless-steel, and bottom adiabatic plate. Rubber sample was packed in the cavity with 30 mm high, and the cavity had tapers in order to remove the sample quickly from the mold. The upper and bottom dimensions of the cavity were 104×104 mm^2 and 140×140 mm^2, respectively.

Energy transfer in the rubber was predominantly one-dimensional, transient heat conduction, and vertical temperature profile and temperature history were measured. Five cartridge heaters with 9.4 mm diameter were embedded in the heating plate. At the bottom surface of the heating plate, nine semi-circular grooves with 0.75 mm radius were machined. Five 0.5-mm-dia Type-K sheathed thermocouples were embedded in the grooves immediately below the heaters to control the heating surface temperature. Four 0.5-mm-dia type-E sheathed thermocouples were located between the type-K thermocouples as the wall thermocouples. The bottom adiabatic surface with thickness of 6-mm consisted of Teflon and silicon rubbers, and a counter heater was attached to the surface to maintain the adiabatic condition. In addition, the heating plate and the side wall was spaced by a Teflon sheet to minimize heat conduction between them, and the counter heaters were attached to the side wall, and heated the wall if necessary.



(a) Mold and rubber sample, front view



Test pieces for image analysis or swelling test

(b) Rubber sample, Cross section A-A

Fig. 1. Experimental apparatus.

To measure the through-thickness temperature profile along the central axis in the rubber, type-J thermocouples were located from the heating surface (x = 0 mm) to the adiabatic surface (x = 30 mm) with an interval of 5 mm as the rubber thermocouples. In the plane at x = 25 mm, lateral rubber temperature distributions were measured by four thermocouples located at 20 mm away from the central axis (positions of E, W, S, and N shown in Fig. 1). All the thermocouples were led out through the mold and connected to the data logger, and the temperature outputs were subsequently recorded with a resolution of 0.1 K.

Major components of the rubber were elastomer, curing agent and blowing agent. The elastomer included 70 wt% styrene-butadiene rubber (SBR) and 30 wt% natural rubber (NR). The peroxide and the D-nitrosopentamethylenetetramine (DPT) were used as the curing agent and the blowing agent, respectively. To locate the rubber thermocouples at the prescribed positions, rubber sheets with thickness of 2, 3 and 5 mm were superposed appropriately, and the thermocouples were sandwiched between them. The weight of the sample packed in the mold was (449 ± 8) gr.

Experiments were conducted under the condition of the heating wall temperature T_M of 140 ℃. Heating time τ_H was changed in several steps from 210 to 330 minutes in order to study the dependencies of the cell structures on the crosslink density. AC 200 volt was applied to the cartridge heaters at the beginning of the experiments to attain the quick rise of the heating wall temperature to the prescribed value. After the temperature was reached to the prescribed value, the heater voltage was reduced in the range of 80 to 120 volt. The temperature of the heating wall was controlled using five electronic controllers to which the type-K thermocouples were connected and the heaters were controlled individually.

During the experiments, the rubber was pressurized through the bottom adiabatic wall using a hydraulic jack. At the predetermined heating time was reached, removal of the hydraulic jack led an instantaneous drop of the rubber and the blowing started under the atmospheric condition. After the blowing was ter-

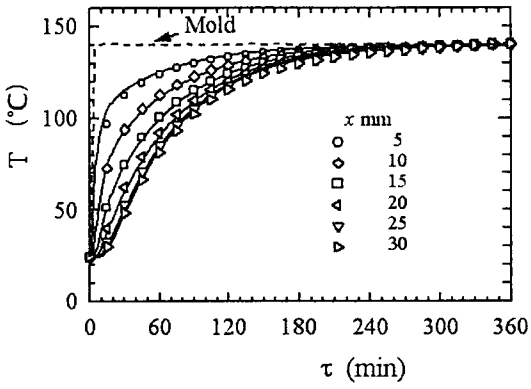
minated, the rubber was immersed in ice water and a rubber sheet with 3 mm thick parallel to the cartridge heater was sliced and three band plates, each having 3 mm width × 30 mm long, were cut out to analyze the cell structure. The sample images were extended ten times using a microscope, and the cell structures were analyzed.

Crosslink density was evaluated from the measured results of the swelling test with toluene. Gas bubbles in the rubber can be considered to give possible, serious error to the swelling test, thus the compounded rubber without the blowing agent was used. The experimental cure methods were the same as that for the blowing experiments. Small pieces with $3\times3\times3$ mm^3 for the swelling test were cut out at the horizontal planes of x= 5, 10, 15, 20, and 25 mm {shaded regions in Fig. 1(b)}. The crosslink density was estimated from the Flory-Rehner equation using the measured results of the swelling test.

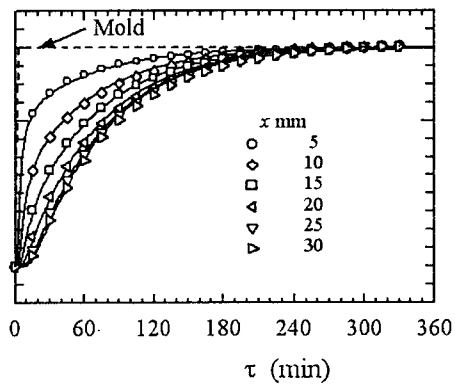
3. Experimental results and discussion

3.1 Temperature Profile

Fig. 2 compares the temperature profiles in the rubber sample, where Fig. 2(a) is the results for the cured rubber and Fig. 2(b) the compounded rubber. Dashed lines show the heating wall temperatures T_M, and the symbols ○, □, ▽ etc. are the rubber temperature T_R. In Fig. 2(a) for the cured rubber, a quick temperature rise with large temperature distribution exists near the beginning of the heating, then these characteristics decrease with the increase of time, and the rubber temperature reached the heating wall temperature at τ= 290 min. Solid lines in Fig. 2(a) shows the numerical results for one-dimensional, transient heat conduction. Comparison of the measured and numerical results shows an excellent agreement except in the vicinity of x = 30 mm at large τ. In addition, the measured temperatures at the positions E, N, S, W in Fig. 1 agreed to that on the central axis in the range of -0.3 to 0.7 K. The fact in Fig. 2 confirms that the one-dimensional, transient heat conduction is predominant, at least, in the middle, horizontal 40×40 mm^2 region in the rubber. In Fig. 2(b) for the compounded rubber, similar temperature field can be observed as that in Fig. 2(a). This implies that the effect of heat generation due to cure reaction may be negligible in the present work.



(a) Cured rubber



(b) Compounded rubber

Fig. 2. Measured and predicted temperature profile.

3.2 Observation of Cell Structure

Cell structure in foam rubber is usually divided broadly into open-cell foam and closed-cell form. Fig. 3 is the binarized images of the cell structure for seven steps of heating time. An overall inspection of Fig. 3 shows clear dependencies of cell characteristics on both the heating time and the distance from the heating surface. The result for τ_H = 210 min., closed-cell form can be observed only near the heating surface, while the open-cell form is pre-dominant over the rubber. For τ_H = 230 min., on the other hand, the open-cells can be observed far from the heating surface, and the closed-cell form is predominant along the sample. Result for τ_H = 260 min. shows closed-cell foam across the rubber, except near the heating surface where no foam exists. In the region τ_H < 260 min., graded structure can be observed clearly, while for τ_H = 330 min., small and uniform closed-cells distribute across the sample.

3.3 Analyses of Cell Structure

Fig. 4 shows the results of the image analyses for the results of Fig. 3. The analyses were performed at x = 5, 10, 15, 20 and 25 mm with a square 3×3 mm^2 samples, where x is the distance measured from the heating surface. In Fig. 4, ν is the crosslink density, N is the number of bubbles observed in 3×3 mm^2 sample, α is the porosity, A is the average bubble area. The crosslink density ν decreases monotonically along x, and at the same x, ν is larger for longer τ_H. The distribution of N for $\tau_H > 230$ min. shows a slight convex upward. The values of N range from 20 to 40, and the effect of τ_H seems to be small as compared to ν. The porosity gradient $d\alpha/dx$ for $\tau_H > 220$ min. decreases with the increase of τ_H, and the α value for τ_H=330 min. is almost uniform along the x. The average bubble area A determined from N and α shows almost similar trends as that for α.

The considerations for Figs. 3 and 4 suggest that the values of N, α and A may be controlled by ν. Fig. 5 shows the dependencies of N, α and A on ν. Number of bubbles N gradually decrease with increasing ν. Porosity α and average bubble area A decrease monotonically in the region $\nu < 0.7\times10^{-4}$ mol/cm^3, while the decreases in the region $\nu > 0.7\times10^{-4}$ mol/cm^3 are negligibly small. Excepting the data for $\nu < 0.7\times10^{-4}$ mol/cm^3, all the data may be uniquely correlated by ν.

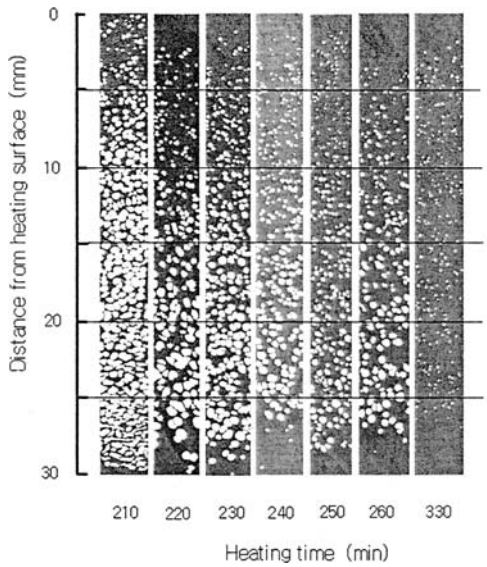


Fig. 3. Binarized images of cell structure for τ_H = 210~330 min.

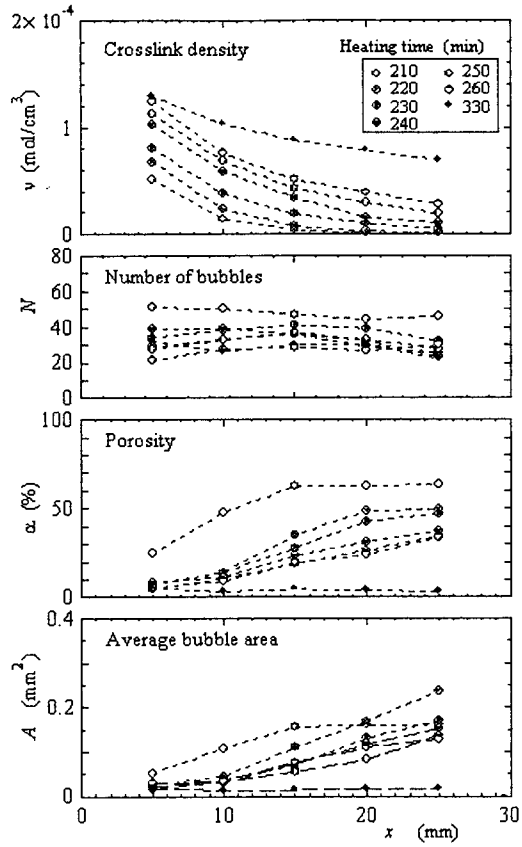


Fig. 4. Effects of τ_H and x on ν, N, α and A.

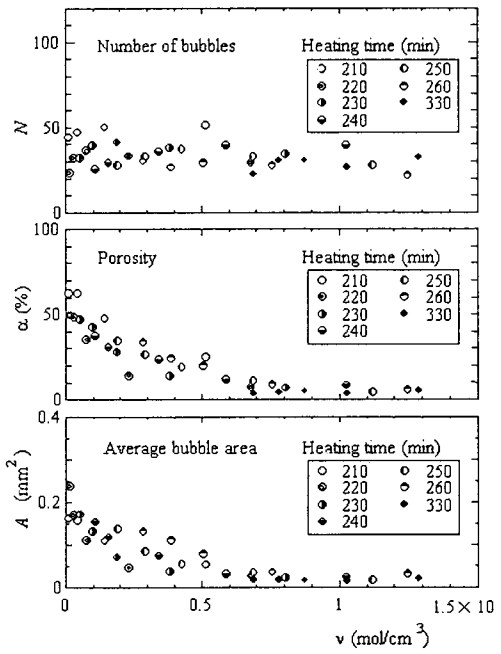


Fig. 5. Dependencies of N, α and A on ν.

4. Conclusions

An attempt has been conducted to observe cell structure during the cure process of SBR/NR foam rubber. Results showed that the cell structure changed depending on both the heating time and the position from the heating surface. Main findings are as follows.

(1) Two extreme cell structure, open-cell foam, and closed-cell foam, were clearly observed.

(2) Porosity increased with increasing the distance from the heating surface, while the increase rate was smaller for longer heating time.

(3) Number of bubbles, porosity and average pore area may be correlated uniquely by crosslink density for the region larger than 0.7×10^{-4} mol/cm^3.

(4) Graded structure was formed for the heating time less than 260 min.

Accumulation of experimental data sets is required to clarify the mechanism of the process.

References

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