Small-angle Neutron Scattering Study on Nonuniformity in Natural Rubber Vulcanizates

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Natural rubber (NR) is one of the most important industrial materials among many polymeric ones. Generally, NR products are prepared by sulfur cross-linking reaction, and the network has been thought to give characteristic properties as rubber materials. However, it is difficult to elucidate the microscopic structure of sulfur cross-linked NR (S-NR) due to non-rubber components in NR such as proteins and lipids [1] in addition to sulfur cross-linking reagents, intermediates and by-products in the reaction [2]. In this study, we applied small-angle neutron scattering (SANS) experiments to S-NR and the results were compared with those of sulfur cross-linked isoprene rubber (S-IR) [2] in order to reveal characteristics of network structure in S-NR.

S-NR was prepared by milling with cross-linking reagents and heat-pressed for curing at 140 °C. SANS experiments were carried out at SANS-U (C1-2), JRR3M in JAEA (Tokai). The wavelength was 7 angstrom. The sample-to-detector distances were 1.00 and 8.00 m. The scattered intensity was collected with an area detector and then circularly averaged. Swollen samples in deuterated (D-) toluene were subjected to SANS measurements.

The following observations were obtained when S-NR and S-IR were prepared under the same reaction conditions for cross-linking: (i) In S-NR, the network inhomogeneity was detected as shown in Fig.1, which was speculated to be due to the presence of poor and rich phases of cross-linking sites and aggregates of non rubber components. (ii) The scattering curves of swollen S-NR in D-toluene were successfully reproduced by the Squared-Lorentz and Lorentz functions. (iii) Both mesh size and domain size (a scale of inhomogeneity ascribed to the rich phase and the aggregate) in S-NR were smaller than those of S-IR. Since NR contains fatty acids, the network formation by zinc salts of stearic acid and fatty acids resulted in the smaller mesh size in S-NR than in S-IR. Larger consumption of zinc oxide in the network formation of the matrix was supposed to bring about the smaller domains in S-NR than in S-IR. These results will be discussed with the mechanical and thermal properties of S-NR and S-IR.

References

[1] T. Karino et al., Biomacromolecules, 8, 693(2007).
[2] Y. Ikeda et al., Macromolecules, 42, 2741(2009).

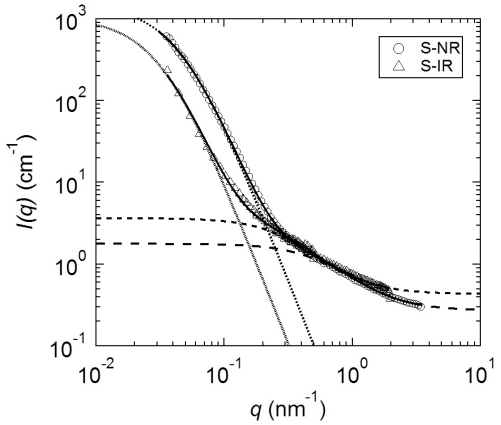

Fig. 1. SANS profiles of S-NR and S-IR. The lines show the results of fitting analyses. Mesh size and domain size were 1.65 nm and 21.7 nm for S-NR and 3.19 nm and 30.9 nm for S-IR, respectively.