Evaluating crosslinking structures by small-angle neutron scattering measurement of sulfur crosslink-controlled rubber

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Improving the physical properties of rubber products requires controlling the homogeneity of the network structures. However, the crosslinking structures of sulfur cross-linked rubber are typically inhomogeneous. In our experiment, two types of rubber samples, an effective vulcanization (EV) system (SBR1, SBR 100/ZnO 3/stearic acid 1/tetramethylthiuram disulfide 4) and a conventional vulcanization (CV) system (SBR2, SBR 100/ZnO 3/stearic acid 1/sulfur 2/1,3-diphenylguanidine 4), with controlled sulfur crosslinking structures were prepared. We evaluated the changes in the inhomogeneous structure and network structure of each rubber sample swollen by toluene-d_8 upon each sulfide bond cleavage using small angle neutron scattering (SANS) measurements and fitting of their profiles. SANS measurement was performed at the SANS-J spectrometer at JRR-3[1]. Fig. 1 shows SANS profiles of sample rubber. For CV system, sulfur bond cleavages—disulfide bonds and polysulfide bonds—resulted in a stepwise decrease in the highly crosslinked domain size and a stepwise increase in the network mesh size. In contrast, the network mesh size did not change much due to the bond cleavage for EV system. This is consistent with the results of crosslinking density measurement by the swelling method. This work will enable the development of rubber products with precisely controlled network structures.

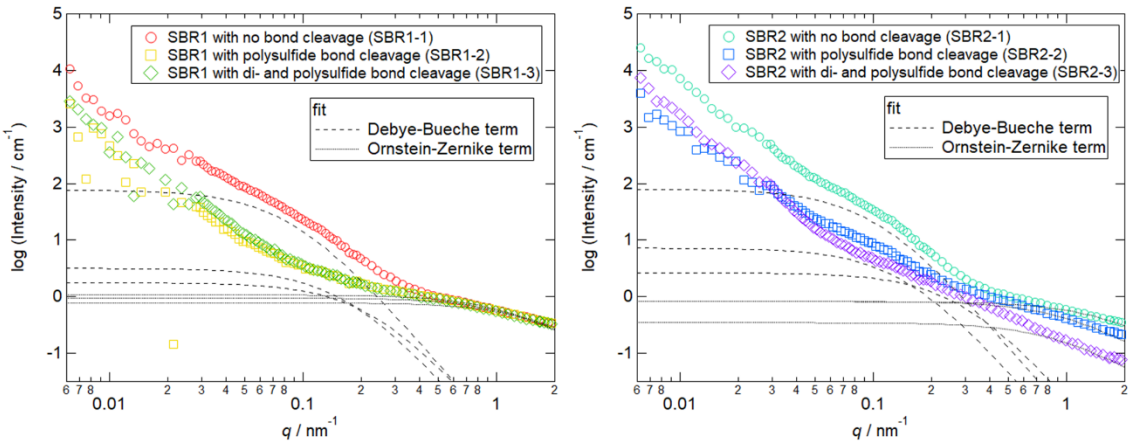

Fig. 1. SANS profiles of rubber samples; (left) SBR1, (right) SBR2. Dashed lines indicate Debye-Bueche term and dotted lines indicate Ornstein-Zernike term, which are obtained by curve fitting of SANS profiles.

References

[1] Kumada *et al.*, *J. Appl. Cryst.*, **2023**, *56*, 1776.

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