XAFS Analysis of Trace Metal in Metal/Rubber Adhesion Layer in Tire

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The rubber for adhesion with steel wire coated by brass in tire contains the Co salt of fatty acids for improving the adhesion property. To investigate the chemical information of the Co species near the brass plate as a model in cured rubber, the Co K edge XANES measurements have been performed. The XAFS imaging technique was applied to evaluate the spatial difference in the Co species.

As a Co salt of fatty acid, 10 phr (9.1 wt%) of cobalt(II) stearate was mixed with natural rubber, and it was further mixed with carbon black, an oxygen scavenger, and zinc oxide at 150 °C. Furthermore, sulfur and a vulcanization accelerator were mixed at less than 100 °C. The rubbers embedded with a brass plate (5 mm × 5 mm × 0.5 mm thickness, Cu : Zn = 65 : 35) were vulcanized at 150 °C by varying the process times. The XANES imaging measurements were carried out at the Co K edge at BL-4 of the SR Center of Ritsumeikan University.

Figure 1 shows the transmitted X-ray intensity observed for a rubber sample cured with a brass plate for 8 min at 150 °C. The XANES spectra at three regions (I1–I3) depicted in Fig. 1 were measured by scanning the X-ray energy, and the observed XANES spectra are given in Fig. 2. The XANES spectrum at the I1 region showed the sharp peak at *ca.* 7720 eV, and it was similar to the Co(II) species containing the O atoms. The peak intensity was weakened according to the distance from the brass plate, and the absorption edge shifted to the lower energy. The spectrum at the I3 position was similar to that of Co(II) sulfide.

Figure 3 shows the chemical state map of the cured rubber with a brass plate. The Co(II) species were assigned to either the compound containing S (blue) or O (yellow) on the basis of the XANES spectra. It was found that the latter species was distributed in the vicinity of the brass plate. It means that the sulfurization of the Co salt of fatty acid tended to be slow at the area near the brass plate.



Fig. 1 The transmitted X-ray intensities measured at the Co K edge. A brass material was located at the bottom center. Three regions (I1–I3) were used to analyze the XANES spectra.



Fig. 2 The spatially resolved XANES spectra of a rubber sample cured for 8 min at 150 °C. The labels (I1–I3) indicate the three regions shown in Fig. 1.



Fig. 3 Chemical state map of the rubber sample at the near area to the brass obtained by the XAFS imaging measurement.