Development of physical property measurement of molten metals by SR X-ray diffraction

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X-ray diffraction from molten metals gives us useful information such as pair distribution function which is concerning material properties. In this trial-use (TU) experiment, bulb-shape sample was floated by gas and heated by laser or arc discharge as shown in Fig.1. Typical sample was SUS304 with a diameter of 2.4mm. Used gas was Ar with flow rate of 150ccm. CO2 laser (SYNCRAD 100) was controlled up to power of 40W. X-ray diffraction data were recorded on Imaging Plate (IP) in 5min.

Fig.1 Schematic of experimental setup.

Figure 2 shows a CCD image of SUS304 heated by laser. As shown in this figure, only the sample ball became bright when heated. Laser heating works well with the floating system.

Fig. 2 CCD image of heated sample ball (SUS304).

Figure 3 shows the diffraction data taken for heated SUS304. It is found that diffraction peaks shifted to smaller diffraction angle (2θ) and reduced their height when heated. This result indicates that heated metal sample was expanding and disordering but not completely melted.

Fig. 3 X-ray diffraction data taken from SUS304 for room temperature (L0) and two CO2 laser energies (L1 and L2).

Temperature of sample must be measured for further experiment.

Analysis of coordination reactions of various vulcanization accelerators in the process of the curing reaction

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Introduction

The vulcanized rubber is obtained by applying heat to the raw rubbers dispersed the vulcanization agents such as sulfur, accelerators (mainly zinc complexes) and the zinc oxide. Accelerator forms the zinc complex by reacting the zinc oxide, this accelerates the vulcanization. It is probably that the sulfur insert into the Zn-S bonds of zinc complex, but this mechanism is not known enough.

In this study, we had investigated about the coordination reactions of a vulcanizing accelerator in a process of the curing reaction by using XAFS technique.

Experiments

The XAFS spectra were carried out in the first batch of BL19B2 in SPring-8. Si(311) double crystals were used to obtain a monochromatic X-ray beam. The Zn K-edge spectra were recorded in transmission mode at 300K. Fourier transformation was performed on k1-weighted EXAFS oscillations in the range of 3-14 Å-1.

The bis(mercaptobenzothiazole) Zn(II) salt (MZ) and the polybutadiene was chosen, as a rubber and Ac. We measured the XAFS spectra about MZ and the rubbers cured for 5, 10, 15, 30, 60min, respectively.

Results

Figure 1 shows the Zn k-edge XANES spectra, and Figure 2 shows the Fourier transform of k1-weighted Zn K-edge EXAFS spectra of vulcanization rubbers and MZ. We obtain slightly deferents from these spectra. Further analysis is now in progress.

Figure1. The XANES spectra of (a) MZ and (b) - (f) rubbers cured for 5, 10, 15, 30, 60min.

Figure2. The EXAFS spectra of (a) MZ and (b) - (f) rubbers cured for 5, 10, 15, 30, 60min.