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Investigation of chemical state of Pb species after Mechanochemical treatment of fly ash by means of XAFS

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Introduction

Recycling of the incineration residues such as bottom and fly ash must be accomplished because of limited capacity of the existent waste disposal sites, although dioxins and heavy metals in the incineration residues, especially fly ash, cause environmental concerns. We focus on mechanochemical (MC) treatment as an effective method for recycling fly ash as a cement material. Previous researches have confirmed the degradation of dioxins in fly ash by MC treatment using CaO as an additive and the inhibition effect on the leachability of heavy metals from fly ash by MC treatment and subsequent cementation [1]. In order to account for the inhibition effect of heavy metals, the chemical state after the MC treatment must be revealed. It is impossible to determine chemical speciation of heavy metals in fly ash with physical methods such as XRD, XPS because of its low concentration, whereas X-ray adsorption fine structure (XAFS) spectroscopy allows heavy metals speciation in diluted system to be performed [2]. So, we investigated chemical state of lead (Pb) species after MC treatment of fly ahs by XAFS method.

Experimental

MC treatment was performed by milling fly ash and the additive CaO using a planetary ball mill (Pulverizette-7, Fritsch, Germany) with a pair of pots (45 cm3) in each of which 7 balls (15-mm diameter) were arranged. 1 g of the fly ash and 4 g of CaO were charged into each pot together under atmospheric condition, and then the planetary ball mill was operated for 4 h at 700 rpm.

After MC treatment, the treatment residue was used for X-ray absorption near edge structure (XANES) spectrum analysis by XAFS method using the BL-12C system in order to determine Pb species. In XAFS measurement, Pb speciation was carried out by the 19-element semiconductor detector at the fluorescent mode at the energy range around 13 KeV equivalent to Pb-L III-absorption edge.

As the reference materials, Pb, $PbCl_2$, PbO, Pb_3O_4 were measured by Si (111) monochromator at the transmission mode.

Results and Discussion

The fly ash treated mechanochemically with CaO for 1, 2 and 4 h were analyzed by XAFS method, and fig. 1 shows the obtained XANES spectra. Compared with XANES spectra of reference materials, spectrum of fly ash is likely in agreement with that of $PbCl_2$. In addition, relative energy position of the edge peak on XANES spectrum of Pb in the fly ash was close to that of $PbCl_2$. This indicates that $PbCl_2$ may be the major species of Pb in the fly ash.

On the other hand, spectra of fly ash treated mechanochemically (1h-MC, 2h-MC and 4h-MC in fig. 1) were obviously different from that of fly ash, indicating that Pb speciation in fly ash was changed by MC treatment. The treated fly ash showed the similarity in spectrum with Pb_3O_4 and the relative energy position of the edge peak on the XANES spectra of the fly ash treated for 1 h and 2 h was same as that of Pb_3O_4. These results confirmed the production of Pb_3O_4 and its further transition by MC treatment of fly ash. Since Pb_3O_4 is insoluble in water, the production of Pb_3O_4 is one of possible explanation for the inhibition of Pb leaching by MC treatment.

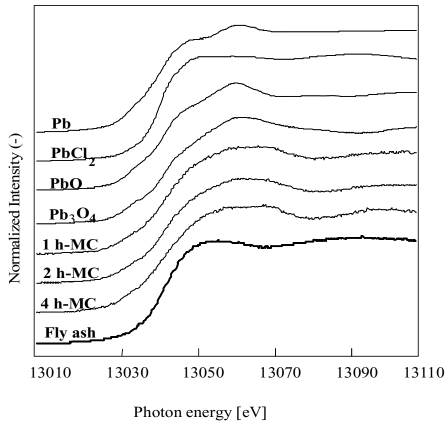

Fig. 1 XANES spectra of untreated fly ash and the reference materials measured by XAFS method.

References

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