The chemical structure for Ag, Cu, Zn and Pb in riddling ash of MSWI residue

H. Yamamoto (636), K. Ohshita (9062) and M. Takaoka (6056)
1) Engineering Research Center, JFE Engineering Corporation, Minamimisukidai 1-1,
Kawasaki 210-0855, Japan
2) Dept. of Environmental Engineering, Kyoto University, Sakyo-ku, Kyoto 606-8501,
Japan

We found that riddling ash that was contained from 1% to 10% of Cu, Zn and Pb, and from 10ppm to 1000ppm of Au and Ag. Such valuable metals was hopped in recycling for raw-material of non-metal industry. However, most of ashes of MSWI residue were landfilled because of its low quantities of valuable metals. In this study, XAFS was used to analyze chemical state change of valuable metals in riddling ashes that was treated by different oxidation state and heat conditions.

Four riddling ashes (Ag 300ppm, Cu 4000ppm, Zn 3000ppm, Pb 1000ppm) was collected from a stoker type furnace in Japan. There were four sampling areas under furnace. Several sampling area were called that "dry-burning for MSWI residue area", "burning for MSWI residue area", "aging ash area1" and "aging ash area2". Then, it was able to compare that the changes of chemical state for valuable metals in different heat condition and oxidation state.

The XAFS measurements were carried out at the BL01B1, SPring-8 with a two-crystal and Si(111) monochrometers. The Ag K, Cu K, Zn K and Pb L3 XAFS spectra were measured in the fluorescence mode using a Ge 19-elements solid-state detector. The spectra for standard materials were measured in the transmission mode.

Fig. 1 shows Ag K-edge absorption near edge structure (XANES) spectra for the four riddling ashes for different area, as well as the spectra for standard materials. A spectrum form of riddling ash of dry-burning area is similar to that of metal-Ag, and spectra form of another riddling ashes were different metal-Ag and other standard materials.

In the case of Cu, Zn and Pb, dry-burning area and burning area were more similar to that of metal-Cu, Zn, Pb, and aging area 1 and 2 were similar to that of CuO, ZnO and PbO.

Fig. 2 shows Pb L3 X-ray absorption near edge structure (XANES) spectra for the four riddling ashes for different area. It assumed that dry-burning and burning area were high temperature more than 900°C, and low oxide concentration state under 1%, since Ag, Cu, Zn and Pb in MSWI residue were reduced in burning area.

Small single crystal structure analysis using new monochromator and low-temperature vacuum X-ray camera

N. Yasuda (4977), Y. Oneda (1167), A. Nishawa (4386),
K. Takeshita (399), Y. Ozawa (1237) and K. Toriumi (3157)
1) Japan Synchrotron Radiation Research Institute (JASRI / SPring-8)
2) Japan Atomic Energy Research Institute (JAERI / SPring-8)
3) RIKEN / SPring-8
4) Graduate School of Material Science, University of Hyogo

The crystal and molecular structures are key information for understanding the chemical and/or physical properties of materials. In order to explore new functional materials, it should be essential to determine whether the relevant molecules take desired structures, conformations, and also intermolecular interactions in crystals.

Single crystal X-ray analysis is mostly conventional and powerful tool for the crystal structure determination. However, the single crystal analyses are frequently encountered difficulties because of extremely small sizes of crystal specimens even though high requirement of their structural information.

High intense synchrotron radiation should enable us to perform the crystal structure analyses of such very small crystals with several micrometers.

In previous experiment, two cydine crystals with dimensions 15.0 x 11.7 x 10.0 µm³ and 6.7 x 5.0 x 3.3 µm³ were analyzed using rubbed monochromator in BL02B1 beamline. The determined molecular structure and accuracy for former crystal were comparable to those for laboratory data (crystal dimensions: 440 x 260 x 240 µm³). For the data set of latter crystal, although the crystal structure could be solved by the direct method, all non-hydrogen atoms were refined isotropically and hydrogen atoms located geometrically.

In order to obtain more brilliant synchrotron X-ray beam for structure determination of smaller crystals with high accuracy, Si(111) direct cooled monochromator and monochromated X-ray were installed optical system in BL02B1 beamline and proved to focus synchrotron X-ray beam into narrower area.

Before bending monochromated X-ray beam, all orientation parameters of monochromator were refined without focusing mirror. The beam profile at 15 keV with bended monochromated X-ray is shown in Figure 1(a). The FWHM of focused beam width were about 200 – 300 µm in the energy range 12.4 – 70 keV. Combining the monochromator and focusing mirrors, beam size was about 220 (width) x 160 (height) µm² at 17keV as shown in Figure 1(b). The beam intensity was estimated at least 20 times than that of flat monochromator.

Although antiscattering bend must be reduced to obtain smaller size beam, this focused beam will be useful to determine the structures of microcrystal with low-temperature vacuum X-ray camera installed in BL02B1 beamline by the long-time intensity measurements.

Figure: Profiles of focused X-ray beam: (a) with monochromated monochromator at 15 keV, (b) with monochromated monochromator and focusing mirrors at 17 keV. TC1 slit size are 40 (width) x 1 (height) mm², respectively.