XANES Spectra of Light Elements, Li, B, C, and P Measured at the New BL-2

Masato Tatsumi[1], Kazunori Takada[1], Akiyuki Nishimura[1], Chihiro Yogi[1], Tomoe Sanada[1], Koji Nakanishi[2], Shingo Ikeda[1], Kazuo Kojima[1], Iwao Watanabe[2], Shigero Ikeda[2], Toshiaki Ohta[2]

1. Introduction

In recent years, light-element based materials such as lithium-ion batteries of high energy and power and advanced carbon materials (for example, carbon nanotubes and carbon fiber reinforced plastics) have attracted much attention in industry, and research and development of those materials have actively been carried out. Analysis of XANES spectra is quite effective in clarifying electronic states and local structure of the materials, and knowledge about the relation between the structure and function of the materials will lead successfully to their higher functionality.

Here, we report XANES spectra of Li K-, P L-, B K- and C K-edges for some standard samples measured at the new BL-2.

2. Experimental

All samples were completely ground into fine powder and fixed on a sample holder by using a sheet of adhesive conductive carbon tape. The monochromator at BL-2 is equipped with three gratings having different line-spacings, 300, 600, and 1200 G/mm, and we have tested all of them. Absorption spectra were obtained by the sample current mode (SC), total electron yield mode (TEY), or fluorescence yield mode (FY). FY spectra were recorded by applying so high negative voltage to a retarding grid as to repel any electrons emitted from the sample. Figs. 1 and 2 show the arrangement of an MCP in the sample chamber and the voltage values applied to the electrodes. The spectra were normalized by using a gold mesh I_0 current.

1) Department of Applied Chemistry, Faculty of Science and Engineering, Ritsumeikan University, Kusatsu, Shiga 525-8577, Japan

2) SR Center, Ritsumeikan University, Kusatsu, Shiga 525-8577, Japan

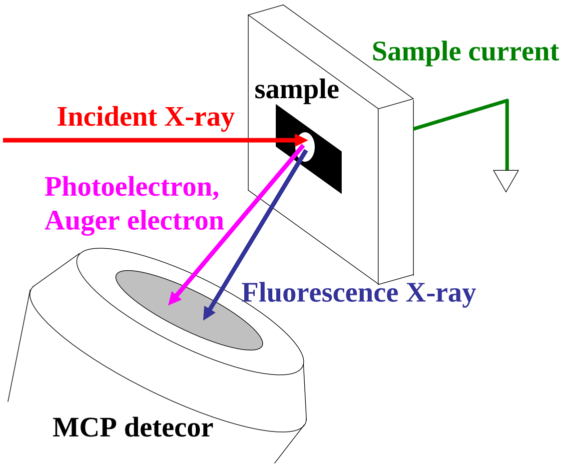


Fig. 1. Arrangement of sample and detector.

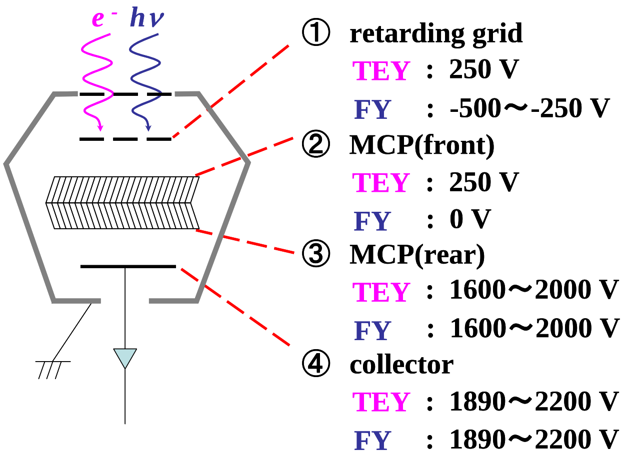
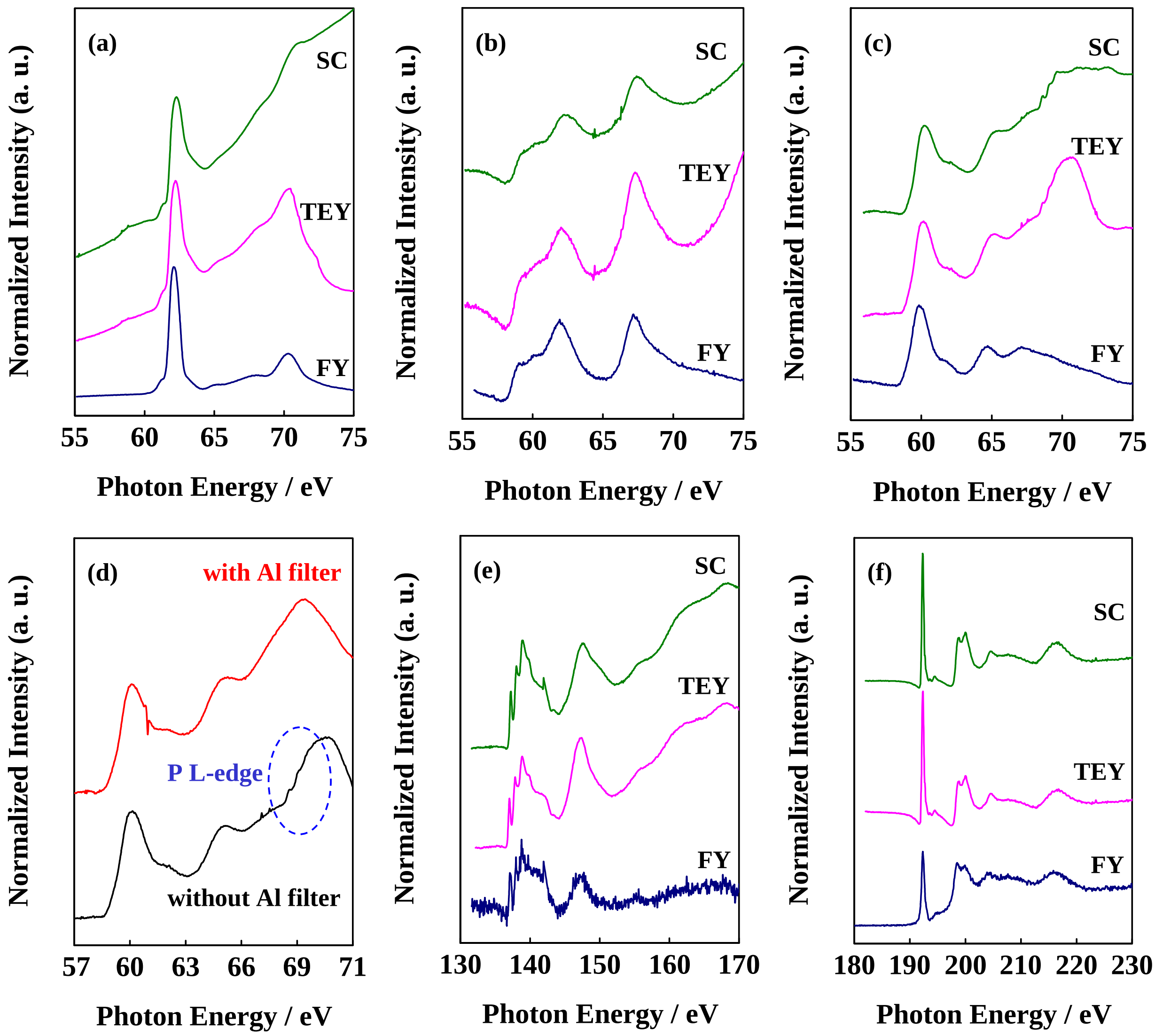


Fig. 2. Setup of the MCP detector.

3. Results

Figs. 3 (a) − (d) show Li K-edge XANES spectra of some lithium compounds. In the LiF spectra (Fig. 3(a)), a very sharp absorption peak and a broad absorption band were observed at 62.3 and 70.5 eV, respectively [1]. The spectral features in SC, TEY, and FY spectra agree well with each other, however, it can be clearly found that S/B ratios in FY spectra are greater than those in SC and TEY spectra. This tendency was found to be the same in other lithium compounds. Since the emission probability of X-ray fluorescence is far smaller than that of Auger electrons for light elements like Li, there is the possibility that the FY method detected luminescence photons [2]. In the TEY spectrum of Li K-edge in Li_3PO_4, there appeared some peaks due to higher-order diffractions from the grating-monochromator, which were successfully removed by inserting an aluminum filter of 150 nm in thickness into the beamline.

XANES spectra of *h*-BN at B K-edge, Li_3PO_4 at P L-edge, and graphite at C K-edge are also shown in Fig. 3. For these samples the FY method gave lower quality spectra, i.e. smaller S/B and S/N ratios.



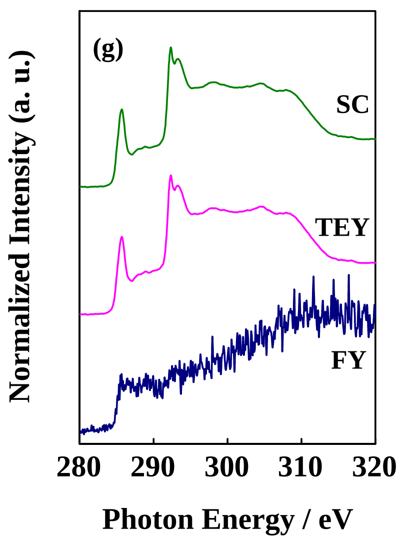


Fig. 3. Li K-edge XANES spectra of (a) LiF, (b) Li_2CO_3, (c) Li_3PO_4, and (d) Li_3PO_4 with and without Al filter, and spectra of (e) Li_3PO_4 at P L-edge, (f) *h*-BN at B K-edge, and (g) graphite at C K-edge.

References

[1] A. Braun et al., *J. Power Sourcs*, 170 (2007) 173-178.
[2] J. G. Zhou et al., *J. Mater. Chem.*, 19 (2009) 6804-6809.