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Quantitative Analysis of a Lithium Ion Battery Cathode Material with X-ray Photoelectron Spectroscopy and Auger Electron Spectroscopy

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Research of Li-ion batteries has been intensively carried out to improve its performance. XPS (X-ray Photoelectron Spectroscopy) and AES (Auger Electron Spectroscopy) are typical analytical techniques for direct detection of Lithium. But these instruments have quite different feature such as analysis area, escape depth of Lithium signal and so on. For the XPS case, the x-ray source of a laboratory system is usually Alkα (which is about 1.5 keV) and MgKα (which is about 1.3 keV). The binding energy of Li1s orbital is around 50 eV, so the Kinetic energy of Li1s photoelectron is at least 1 keV. XPS has two advantages to analyze Li; the photoelectron with energy over 1 keV has a long mean free path, and the region of the Li 1s spectra has very low background [1]. These are the reasons why Lithium is detected easily by XPS. Thus, XPS can provide useful information for Li-ion battery materials. In the charging and discharging process of Li-ion battery, Li ions move between positive and negative electrodes. The movement of Li ions causes the change of the chemical bonding states of the transition metal in order to retain the charge balance of positive electrode. For these reason, XPS is very useful to analyze the materials of Li-ion batteries. An XPS system for laboratory use has been developed for micro area analysis (it is several tens micrometer) [2]. The micro area analysis is suitable for semiconductor samples. But the difficulty of the XPS micro area analysis is to precisely determine the analytical position. Of course it is easy to determine the analytical position of the sample with a clear structure like printed board and an area of discoloration. In the case of powder samples, it is very difficult to determine its analytical position because most powder samples have no specific feature. But in many cases, powder samples have the compositional differences.

At this study, XPS analysis of a Li-ion battery cathode material was performed. The equipment is a JPS-9200 (JEOL) which has a combined electromagnetic and electrostatic input lens system and two apertures inside the input lens. This system makes it possible to change the diameter of the analytical area from 30 μm to 3mm. Fig. 1 shows the result of qualitative and quantitative analysis of an XPS measurement at three different points with a 0.1 mm diameter. Fig. 2 shows the results of qualitative and quantitative analysis of the XPS measurement at three different points with a 3mm diameter. The quantitative data in Fig. 1 (0.1 mm diameter) shows a considerable dispersion, whereas that in Fig. 2 (3mm diameter) shows no such dispersion. Recently it was showed that AES has the great potential to analyze Lithium ion battery materials because AES can detect Lithium directly and AES can analyze chemical state of transition metal elements [3]. And off course AES has the function to measure the back scattered electron image (Fig. 3) not only secondary electron image. The high spatial resolution image contains information about compositional differences, which is quite useful to determine the analytical position even in powder samples. In the present study we will show the secondary electron image and back scattering electron image using SEM (Scanning Electron Microscopy) and the auger spectra measured with a JAMP-9510F (JEOL). We discuss the dependence of XPS, SEM and AES data on analytical positions. We propose the creatively use the XPS and AES analysis for Li-ion battery materials.

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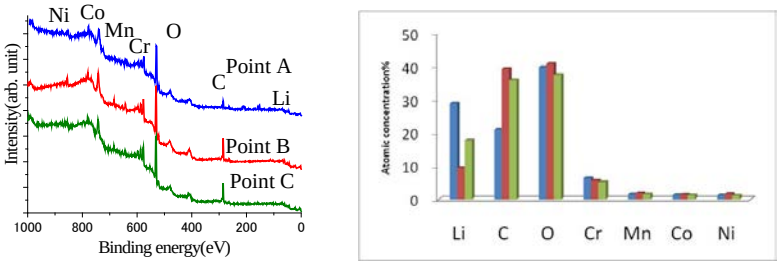

Figure 1 Qualitative and quantitative analysis with a 0.1 mm diameter with XPS

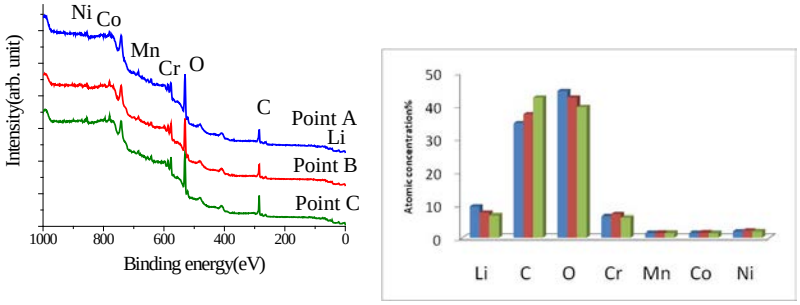

Figure 2 Qualitative and quantitative analysis with a 3 mm diameter with XPS

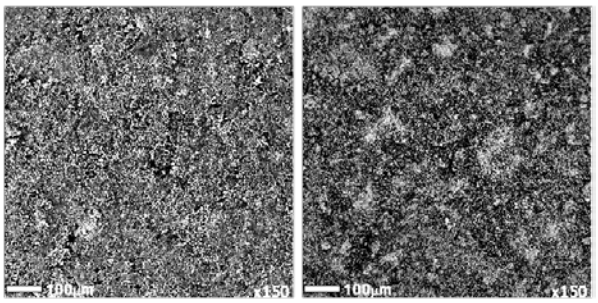

Figure 3 secondary electron image(left)and back scattered electron image(right) of LiB cathode material observed with JAMP-9510F

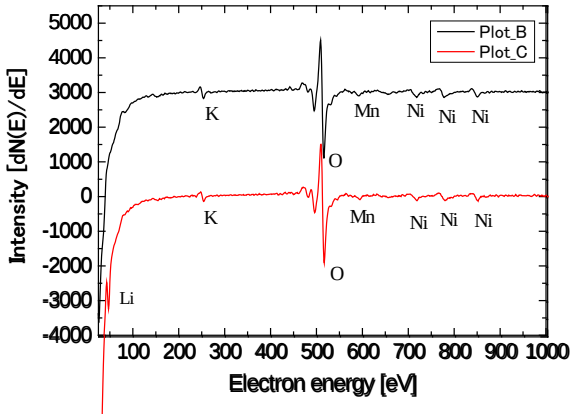

Figure 4 Auger spectra of LIB with JAMP-9510F at dark and bright area in fig. 3

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