

X-ray diffraction analysis of micro-crystals by using Gandolfi camera

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In our previous study (1999B0221-NOD-np), we have reported that powder diffraction pattern of interplanetary (cosmic) dust particles in 10 μ m size was successfully obtained by a combination of a Gandolfi camera and Imaging Plate. Moreover, the crystal structure of a new mineral, kozoite was solved by the Rietveld method using Gandolfi diffraction data. In the present study, we have examined the potential ability of the present technique in material characterization of microcrystals. The samples are cathode materials for lithium ion secondary battery. Their single crystals are extremely difficult to be synthesized in large scale.

The single crystals of Co or Ni substituted LiMn₂O₄ spinel were synthesized for the first time by the present study at our laboratory. Syntheses were carried out utilizing a flux (LiCl + KCl) method at 860 $^{\circ}$ C(max.). The products were characterized by SEM-EDX and a Gandolfi camera utilizing SR. The measurements were carried out at BL39XU using undulator X-ray radiation. The powder diffraction data of each

single crystal were recorded on an Imaging Plate with 50 μ m resolution using a 114.6 mm Gandolfi camera and monochromatic X-ray ($\lambda=1.5423$ or 1.9677 \AA) from Si(111) double crystal.

Powder diffraction data of the products are shown in Fig. 1, which confirms that both crystals have a spinel structure with lattice parameters calculated to be $a=8.1959(17)$ and $8.1975(8)$ for the Co and Ni phases, respectively. SEM photographs of the crystals (Fig.2) show that the products are euhedral crystals with well-developed (111) planes. Their EDX spectra support the substitution of Co and Ni for Mn. From these data, we have concluded that the products are Co or Ni substituted lithium manganese spinel phases, which are attractive cathode materials with high energy densities. Present study demonstrated that this Gandolfi technique is suitable for material characterization of microcrystals and will become an important tool in routine industrial analysis of various advanced materials.

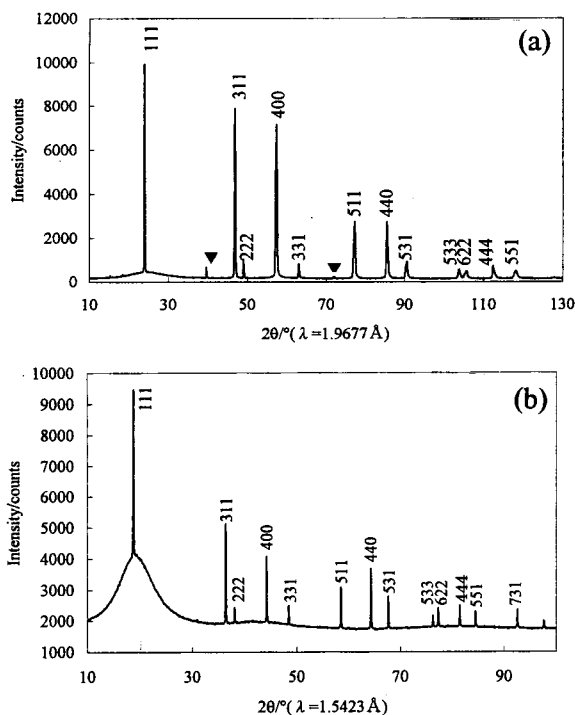


Fig.1 Powder diffraction pattern from a single crystal (a) LiMn_{1.5}Co_{0.5}O₄ and (b) LiMn_{1.5}Ni_{0.5}O₄

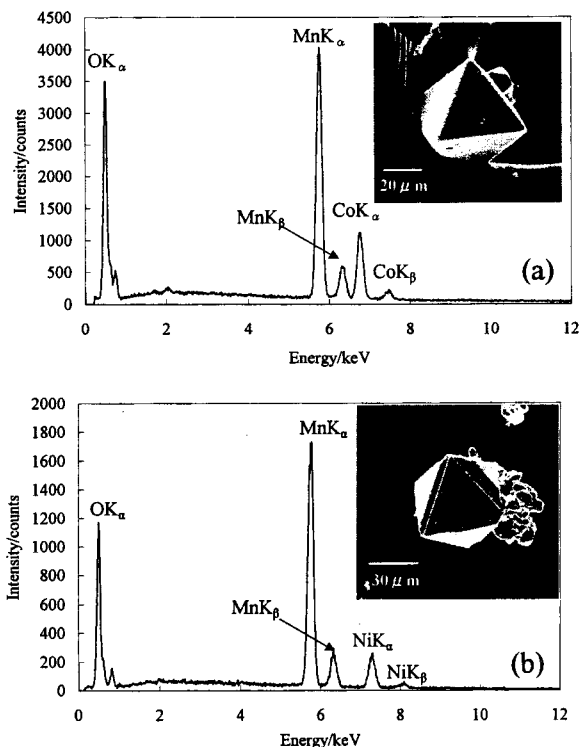


Fig.2 SEM photos and EDX spectra of (a) LiMn_{1.5}Co_{0.5}O₄ and (b) LiMn_{1.5}Ni_{0.5}O₄