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Nanoparticles Synthesis of Lithium Oxide Composite with Refractory Metal for Lithium-Ion Battery Electrodes

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Attractive material processing with thermal plasmas have been widely proposed for the nanoparticles production at a high productivity. This is because thermal plasmas offer unique advantages; high enthalpy, high chemical reactivity, rapid quenching rate, and selectivity of reaction atmosphere.

Lithium metal oxides have attracted many researchers as electrode materials for lithium-ion batteries. Crystal structure control of the lithium metal oxide is essential to enhance the battery characteristics. Layered rock-salt type, spinel type, and olivine type materials are considered as suitable structure of positive electrode material due to its high mobility of lithium ion. In recent, cubic rock-salt type such as Li-Ni composite oxide has attracted attention due to its high stability.

Lithium composite oxide with refractory metals are also expected as candidate of positive electrode material due to its good battery characteristics [1], although no report has been found on nanoparticle synthesis of lithium oxide composite with refractory metals by thermal plasmas. The purpose of the present study is to synthesize cubic rock-salt type Li-Nb-Ni oxide nanoparticles by induction thermal plasmas.

Figure 1 shows a schematic of an experimental setup for the induction thermal plasma. This consists of a plasma torch, a synthesis chamber, and a power supply with frequency of 4MHz. Input power was 20 kW. A powder mixture of Li_2CO_3, Nb, and Ni was used as raw material and introduced into the plasma at a feed rate of 300 mg/min. Mole fraction of raw material was set to Li:(Nb+Ni) = 1:1 to produce cubic rock-salt type. Nb fraction as Nb/(Nb+Ni) was changed in the range from 0 to 1 to clarify the effect of Nb fraction on the crystal structure of the products.

The crystal structure of the synthesized nanoparticles was determined though X-ray diffraction (XRD). The particle morphology was observed by transmission electron microscopy (TEM) and size distributions were estimated. Element mapping of nanoparticles was analyzed by scanning TEM-energy dispersive X-ray spectrometry (STEM-EDS).

The XRD patterns of nanoparticles synthesized at Li:(Nb+Ni) = 1:1 are shown in Fig. 2. Cubic-rock salt type (*Fm-3m*) as target product was successfully synthesized in all conditions. Particularly *Fm-3m* was synthesized in a single phase at Nb/(Nb+Ni) = 0.25, 0.5, and 0.75.

Element mapping images and corresponding dark-field image by STEM-EDS for nanoparticles synthesized at Nb/(Nb+Ni) = 0.75 are presented in Fig. 3. Mapping of Nb, Ni, and O overlapped in the same particles. In other words, Li-Nb-Ni oxide nanoparticles were successfully formed.

The formation mechanism of Li-Nb-Ni oxide nanoparticles were revealed on the basis of the nucleation temperature. The estimated nucleation temperature of Nb was 3076 K, which is highest nucleation temperature in Li-Nb-Ni system. Consequently, Nb nucleates first, then the Li, Li_2O, Ni, and NiO vapors co-condense on the nuclei, forming the Li-Nb-Ni oxide nanoparticle.

Li-Nb-Ni oxide nanoparticles were successfully synthesized by induction thermal plasma. Single phase of cubic rock-salt type as objective nanoparticles was prepared in Li-Nb-Ni system. Thermal plasma synthesis enables to produce attractive electrode materials in lithium-ion battery at high-productivity.

References

[1] N. Yabuuchi, et al., Proc. Natl. Acad. Sci. USA., 112 (25) 7650-7655 (2015).

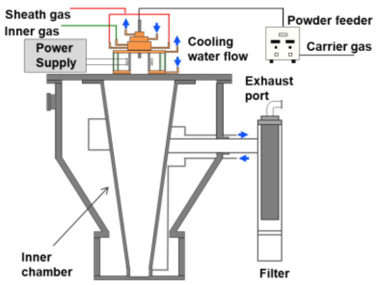

Fig. 1. Experimental setup of induction thermal plasma.

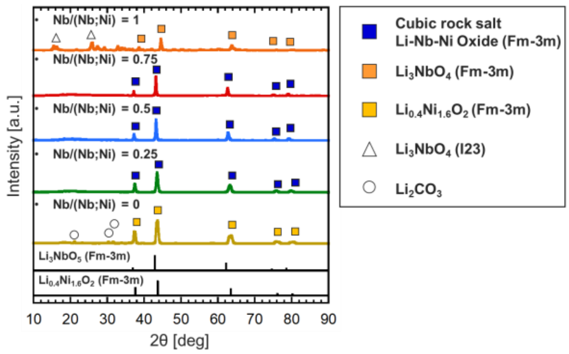

Fig. 2. XRD spectra of prepared nanoparticles at a fixed Li:(Nb+Ni) = 1:1 with different Nb/(Nb+Ni) = 0, 0.25, 0.5, 0.75, and 1.

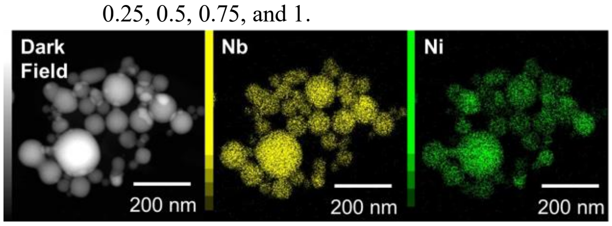

Fig. 3. Element mapping of prepared nanoparticles at a fixed Li:(Nb+Ni) = 1:1 with Nb/(Nb+Ni) = 0.75