APPLICATION NOTE

Air-Protected Analysis of Specific Surface Area and Density of Solid Electrolytes (Oxides/Sulfides) of All-Solid State Battery Cells for Improving the Ion Conductivity of Such Electrolytes

Overview

Recently, all-solid state battery cells have been attracting close attention from the viewpoints of high output density and safety. Small size (large specific surface area) and high density (absence of spore) of the particles constituting the solid electrolytes (oxides/sulfides) are very important in improving ion conductivity. Meanwhile, since each solid electrolyte involves a risk of carbonate precipitation and the release of hydrogen sulfide when exposed to ambient air, it is essential to conduct an evaluation of the specific surface area and density when not exposed to ambient air.

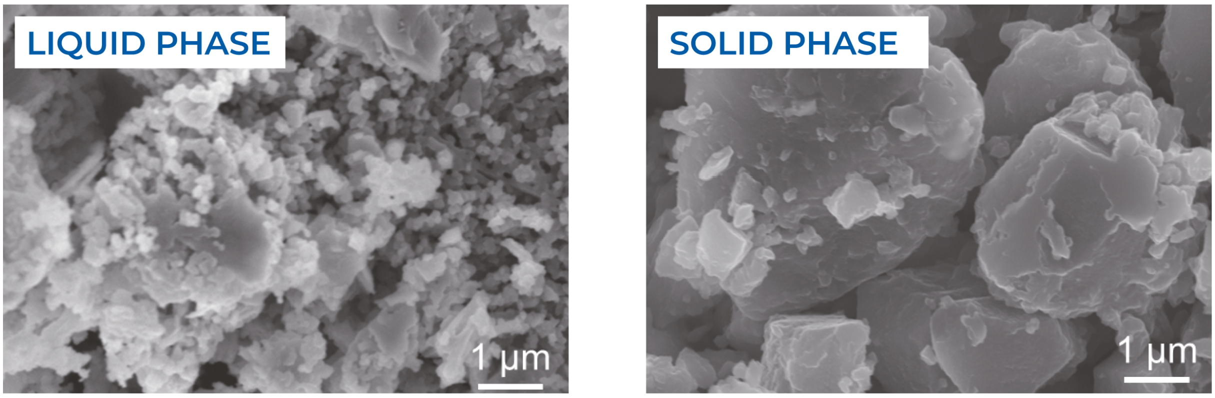

Fig.1 SEM micrograph of liquid-phase / solid-phase synthetic Li_6PS_5Cl

Experiment

Fig. 1 shows a scanning electron microscope (SEM) image of solid Li_6PS_5Cl electrolyte synthesized in the liquid phase and in the solid phase (with the use of a ball mill). Whereas the liquid-phase synthetic material is formed as an aggregation of particles in the 10-100 nanometer range, the solid-phase synthetic material is formed in the submicron size range. Each sample (ca. 0.5 g) was weighed within the NSD capsule inside the special test tube designed for inhibiting air exposure in measurement shown in Fig. 2 within a glovebox under an argon gas atmosphere. After the sample was placed into a test tube fitted with a cock, pretreatment with BELPREP VAC II was conducted at 120°C for 6 hours in a vacuum. Thereafter the N_2 (77.4K) adsorption isotherm was obtained with BELSORP MINI X without being exposed to ambient air.

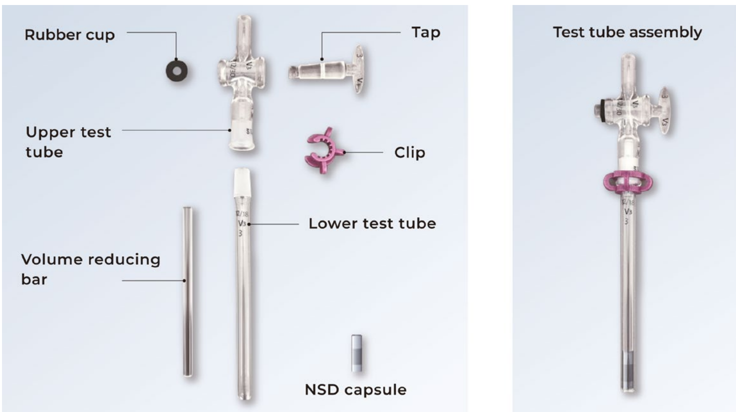

Fig.2 Special test tube for ambient air non-exposed measurements

Results

The adsorption isotherm of the lithium-sulphur sold electrolytes synthesized in the liquid phase and the solid phase (Fig. 3) was a IV and II type isotherm with the BET specific surface area being 20.6 m^2/g and 2.8 m^2/g, resp. When pore distribution (Fig. 3, inner) was evaluated by the BJH method (adsorbing branch), the sample synthesized in the liquid phase had mesopores of about 1-50 nm while the material synthesized in the solid phase was approximately free of pores. Each SEM image shows that the mesopores of the particles synthesized in the liquid phase represented the space between the nanoparticles, and that the particles released in the solid phase were larger in size (micro order) and free of pores, resulting in a smaller specific surface area.
As shown above, a quantitative evaluation of BET specific surface area and density (measured with the special test tube designed to inhibit air exposure in measurements of solid electrolytes for solid-stage battery cells) together with SEM images provides an effective method for examining the ion conductivity mechanism.

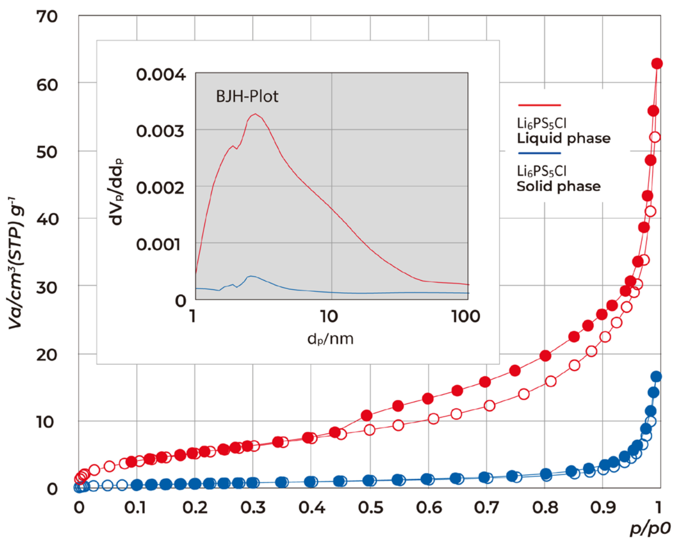

Fig.3 N_2 (77.4K) adsorption isotherm and density evaluation of liquid-phase and solid-phase synthetic Li_6PS_5Cl

Sample / SEM image supplied by Prof. Seiji Tadanaga, Hokkaido University Graduate School of Engineering

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