**Preparation of high lithium-ion conducting Li₆PS₅Cl solid electrolyte
from ethanol solution for all-solid-state lithium batteries**

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Abstract

A Li₆PS₅Cl solid electrolyte was successfully prepared by dissolution-precipitation process *via* ethanol solution. An ionic conductivity of the Li₆PS₅Cl solid electrolyte from the homogeneous ethanol solution was $1.4 \times 10^{-5} \text{ S cm}^{-1}$ at room temperature. LiCoO₂ particles were coated with the Li₆PS₅Cl electrolyte *via* ethanol solution to form favorable electrode-electrolyte interface with a large contact areas. An all-solid-state cell using the electrolyte-coated LiCoO₂ operated as a rechargeable battery and showed the initial discharge capacity of 45 mAh g⁻¹ at 25 °C.

Keywords : All-solid-state battery, Lithium secondary battery, Sulfide solid electrolyte, Liquid phase method

1. Introduction

Lithium secondary batteries have been used for power sources such as electronic equipment because of their high operating voltage, high energy density and long cycle performance [1]. Recently, large-sized lithium-ion batteries are requested for application as power sources for electrical cars and stationary batteries. It is necessary for lithium-ion batteries to have higher energy density and longer cycle performance. Especially, improvement in safety of lithium secondary batteries is essential because organic liquid electrolytes used in conventional lithium secondary batteries have several risks of leakage and ignition.

All-solid-state lithium secondary batteries using inorganic solid electrolytes have been expected as next-generation batteries with higher energy density, safety and reliability [2]. In order to improve the electrochemical performances of the all-solid-state batteries, solid electrolytes with high ionic conductivities are key materials. Recently, $\text{Li}_{10}\text{GeP}_2\text{S}_{12}$ crystal has been reported to show extremely high lithium-ion conductivity of more than $10^{-2} \text{ S cm}^{-1}$ at room temperature [3]. Crystalline phases and lithium-ion conductivities of binary $\text{Li}_2\text{S-P}_2\text{S}_5$ systems have also been studied [4,5]. We reported that $\text{Li}_2\text{S-P}_2\text{S}_5$ glass-ceramic electrolytes prepared using a mechanical milling technique exhibited a high lithium-ion conductivity of $5.2 \times 10^{-3} \text{ S}$

cm⁻¹ at room temperature [6]. All-solid-state cells using the Li₂S-P₂S₅ solid electrolyte operated as secondary batteries [7].

In order to improve the electrochemical performance of the bulk-typed all-solid-state batteries, it is necessary to prepare favorable interfaces between electrodes and solid electrolytes with large contact areas [8-11]. Formation of the interfaces with the large contact areas can provide better ionic conduction path. It has been reported that all-solid state batteries using LiCoO₂ coated with oxide solid electrolytes such as LiNbO₃, Li₄Ti₅O₁₂ and Li₂SiO₃ show a high energy density [12-14]. We have reported that LiCoO₂ particles were coated with Li₂S-P₂S₅ or Li₄GeS₄-Li₃PS₄ solid electrolyte by the pulsed laser deposition method to prepare favorable interfaces between the electrodes and the solid electrolytes [8-10]. The all-solid-state batteries using LiCoO₂ coated with the solid electrolytes worked as rechargeable batteries even at large amounts of LiCoO₂ in the positive electrodes and showed higher capacity compared to those using non-coated LiCoO₂.

Preparation of the solid electrolytes by the liquid-phase method is much simpler and more effective process than the gas-phase method such as pulsed laser deposition (PLD). The favorable interfaces between solids and liquids are easily formed by using the liquid-phase method and then the preparation of the favorable interfaces between

electrodes and solid electrolytes can be achieved by removing the solvents. Recently, synthesis of β -Li₃PS₄ using tetrahydrofuran (THF) has been reported as one way of preparing solid electrolytes by the liquid-phase method [15]. The ionic conductivity of the β -Li₃PS₄ electrolyte was 1.64×10^{-4} S cm⁻¹ at room temperature. It has been also reported that Li₇P₃S₁₁ solid electrolyte, which showed the ionic conductivity of 2.7×10^{-4} S cm⁻¹ at room temperature, was synthesized using 1,2-dimethoxyethane solvent [16]. These solid electrolytes had almost the same ionic conductivities at Li₂S-P₂S₅ solid electrolytes prepared by mechanical milling. However, in these methods, the precursors with precipitates were obtained. It is important that the precursors before drying are liquid-phase materials to prepare homogeneous electrolyte-coatings on electrodes particles with larger contact areas. Recently, we have reported that the Li₂S-P₂S₅ solid electrolytes were prepared using *N*-methyformamide (NMF) solvent [17-19]. 80Li₂S·20P₂S₅ (mol%) glass was prepared by a mechanical milling technique and then dissolved in NMF to prepare homogenous solution. Li₃PS₄ solid electrolyte was prepared from the solution by removing the solvent [17,18]. In addition, the Li₃PS₄ solid electrolyte was directly synthesized from Li₂S and P₂S₅ as the starting materials *via* homogeneous NMF solutions [19]. We have reported that the Li₃PS₄ solid electrolytes can be obtained by the process in dissolution-reprecipitation of 80Li₂S·

20P₂S₅ (mol%) glass and the process in direct synthesis from the starting materials. Moreover, we succeeded in applying the NMF solution to form favorable interfaces between electrodes and electrolytes for all-solid-state batteries, and the all-solid-state cells using LiCoO₂ coated with the solid electrolyte operated as rechargeable batteries without the addition of solid electrolyte and carbon conductive additive particles to the positive electrodes [19]. However, the capacities of the cells were lower than those of the cells using LiCoO₂ coated with the solid electrolytes by PLD because the electrolytes synthesized *via* the liquid-phase showed lower ionic conductivities than those prepared by PLD. In order to improve electrochemical properties of all-solid-state batteries by coatings of solid electrolyte, it is necessary to increase their ionic conductivities.

In order to solve the issue, we have focused on electrolyte compositions and solvents. Argyrodite-type Li₆PS₅X crystals are attracting attention as sulfide-based solid electrolytes with a high lithium-ion conductivity [20,21]. The Li₆PS₅Cl crystal prepared by the mechanical milling technique showed a high lithium-ion conductivity of over 10⁻³ S cm⁻¹ and thus it is a promising candidate of electrolytes prepared *via* a liquid-phase process. The selection of solvents is also important. Ethanol (EtOH) has much higher versatility as a solvent than NMF with a high boiling point of 199 °C. The

use of EtOH with a lower boiling point **should be** effective in preventing side-reactions between the electrolyte coatings and electrode particles during heat-treatment for removing the solvent.

In the present report, $\text{Li}_6\text{PS}_5\text{Cl}$ crystal was prepared by a mechanical milling technique and then dissolved in EtOH to prepare homogenous solution. The solid electrolyte was prepared from the solution by removing the solvent. The solid electrolyte was characterized by X-ray diffraction measurement and Raman spectroscopy. Ionic conductivities of the solid electrolyte were measured by AC impedance measurements. Coating of the solid electrolytes on LiCoO_2 particles was carried out using ethanol solution and the favorable interface between electrode and solid electrolyte with a large contact area was formed. The all-solid-state batteries using the electrolyte-coated LiCoO_2 particles were fabricated and characterized.

2. Experimental

A solid electrolyte $\text{Li}_6\text{PS}_5\text{Cl}$ was synthesized using mechanical milling [20]. Li_2S (Idemitsu Kosan, 99.9 %), P_2S_5 (Aldrich, 99 %) and LiCl (Aldrich, 99.9 %) crystalline powders as the starting materials were mixed in stoichiometric proportions. The total weight of the materials was 1 g. The mechanical milling was carried out using a planetary ball mill (Fritsch, Pulveristte 7) with a zirconia pot (45 mL in volume) and 15 zirconia balls (10 mm in diameter) at room temperature. Rotation speed was 600 rpm and the milling time was 45 hours.

The synthesized $\text{Li}_6\text{PS}_5\text{Cl}$ solid electrolyte powder was dissolved into an anhydrous ethanol (EtOH, Wako, 99.5 %) solvent. After stirring for 24 hours at room temperature, a homogeneous dark brown EtOH solution was obtained. The concentration of the solid electrolyte in the EtOH solution was 10 wt%. The prepared solution was dried at 80 °C under vacuum for 3 hours and then powders were obtained. All the processes were performed in a dry Ar atmosphere.

Raman spectroscopies were carried out with a Raman spectrophotometer (HORIBA Jobin Yvon, Lab-Ram HR-800) to identify the structural units in the original $\text{Li}_6\text{PS}_5\text{Cl}$ solid electrolyte, the EtOH solution and the powder prepared from the EtOH solution. X-ray diffraction (XRD) measurements were performed using an X-ray diffractometer

(Rigaku, SmartLab) with a $\text{CuK}\alpha$ radiation to identify crystalline phases of the original $\text{Li}_6\text{PS}_5\text{Cl}$ solid electrolyte and the powder prepared from the EtOH solution.

An ionic conductivity for the powder obtained from the EtOH solution was measured by AC impedance at room temperature using an impedance analyzer (Solartron, 1260) in the frequency range of 0.1 Hz to 1 MHz. The measurements were performed for a pelletized sample with 10 mm in diameter and about 1.5 mm thick. Au thin films were coated on both faces of the pellets as current collectors. Temperature dependences of ionic conductivity were also performed. All the processes were carried out in a dry Ar atmosphere.

The prepared EtOH solution was mixed with LiCoO_2 particles (D-10, Toda Kogyo Corp.). The weight ratio of LiCoO_2 to the dissolved solid electrolyte was 92.5 : 7.5. LiCoO_2 particles used in this study were coated with LiNbO_3 layer because all-solid-state cells using LiCoO_2 particles coated with oxides such as LiNbO_3 , $\text{Li}_4\text{Ti}_5\text{O}_{12}$ and Li_2SiO_3 showed a high energy density [12-14]. The mixture was dried at 80 °C for 3 hours under vacuum to remove EtOH solvent and then the LiCoO_2 particles coated with the solid electrolyte (SE-coated LiCoO_2) were obtained. The morphology of SE-coated LiCoO_2 was observed using a scanning electron microscopy (SEM, JSM-6610A, JEOL) with an energy dispersive X-ray spectroscopy system (EDX,

JED-2300, JEOL).

All-solid-state cell (In / $80\text{Li}_2\text{S}\cdot 20\text{P}_2\text{S}_5$ (mol%) glass-ceramic solid electrolyte / SE-coated LiCoO_2) was fabricated as follows. Neither solid electrolyte particles nor carbon conductive additives were added to the positive electrode; SE-coated LiCoO_2 particles were only used in the positive electrodes. The $80\text{Li}_2\text{S}\cdot 20\text{P}_2\text{S}_5$ glass-ceramic for solid electrolyte was prepared by mechanical milling and subsequent heat treatment to use as a separator. Indium foil (Furuuchi Chem., 99.999 %, 0.1 mm thickness) was used as a negative electrode. A bilayer pellet (10 mm in diameter) consisting of the SE-coated LiCoO_2 (about 20 mg) as a positive electrode layer and the glass-ceramic electrolyte as a separator layer (80 mg) was prepared by pressing under 360 MPa, and the indium foil was attached to the pellet by pressing under 240 MPa. All the processes were performed in a dry Ar atmosphere.

The charge-discharge measurements for the all-solid-state cell were performed using a charge-discharge measuring device (BTS-2004, Nagano Co., Ltd.) at 25 °C in an Ar atmosphere. The electrochemical tests were performed at a constant current density of 0.13 mA cm^{-2} in the voltage range from 2.0 V to 3.6 V vs. Li-In (corresponding to the voltage range from 2.6 V to 4.2 V vs. Li).

3. Results and Discussion

Fig. 1 (A) shows Raman spectra of the original $\text{Li}_6\text{PS}_5\text{Cl}$ solid electrolyte (a), the ethanol (EtOH) solution (b) and the powder prepared from the EtOH solution after drying at 80 °C under vacuum (c). The Raman bands attributable to PS_4^{3-} units were observed in all the samples. Deiseroth *et al.* has reported that the argyrodite-type solid electrolyte $\text{Li}_6\text{PS}_5\text{X}$ (X = Cl, Br, I) was constructed of PS_4^{3-} units [22]. The powder prepared from EtOH solution thus retained the main structure unit of the original $\text{Li}_6\text{PS}_5\text{Cl}$.

Fig. 1 (B) shows X-ray diffraction (XRD) patterns of the original $\text{Li}_6\text{PS}_5\text{Cl}$ solid electrolyte (a) and the powder prepared from the EtOH solution after drying at 80 °C under vacuum (b). The diffraction patterns of different phases were not detected in the original $\text{Li}_6\text{PS}_5\text{Cl}$ prepared by mechanical milling. Sharper diffraction peaks attributable to $\text{Li}_6\text{PS}_5\text{Cl}$ were observed in the powder prepared from the EtOH solution. The diffraction patterns of argyrodite-typed crystal were observed but those of other crystals were not detected in the powder prepared from the EtOH solution, suggesting that $\text{Li}_6\text{PS}_5\text{Cl}$ crystal can be prepared by dissolution-reprecipitation process via EtOH solution.

Fig. 2 shows impedance spectra of the original $\text{Li}_6\text{PS}_5\text{Cl}$ electrolyte (a) and the

Li₆PS₅Cl electrolyte prepared from the EtOH solution at room temperature (b). An ionic conductivity of the original Li₆PS₅Cl electrolyte was $1.4 \times 10^{-3} \text{ S cm}^{-1}$ at room temperature. The impedance spectrum of the Li₆PS₅Cl electrolyte obtained from the EtOH solution showed a semicircle corresponding to the grain boundary resistance and the total ionic conductivity was $1.4 \times 10^{-5} \text{ S cm}^{-1}$ at room temperature. Activation energies for ion conduction of the original Li₆PS₅Cl electrolyte and the Li₆PS₅Cl electrolyte obtained from the EtOH solution were 22 kJ mol⁻¹ and 33 kJ mol⁻¹, respectively. Lower ionic conductivity and higher activation energy of the Li₆PS₅Cl electrolyte obtained from the EtOH solution were mainly caused by increasing grain boundary resistance. Surface conditions of the obtained Li₆PS₅Cl particles are probably different from those of original ones. Some less-conductive organic components may remain on the surface of electrolyte obtained from the EtOH solution, resulting in increasing grain boundary resistance. The relationship between surface structure and resistance is important for electrolyte prepared *via* the liquid-phase method and will be investigated in detail in the near future.

The EtOH solution was effective in coating active material particles with a large contact area. Fig. 3 shows the scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDX) mapping (Co, P, S and Cl) images of a LiCoO₂ particle

coated with $\text{Li}_6\text{PS}_5\text{Cl}$ solid electrolyte (SE-coated LiCoO_2) from the EtOH solution. The image showed that particle size of the LiCoO_2 was about 10 μm , which was the same as that of non-coated LiCoO_2 . The LiCoO_2 particle was coated with the $\text{Li}_6\text{PS}_5\text{Cl}$ layer because mapping areas of P, S and Cl overlapped the area of Co.

All-solid-state cell using the prepared SE-coated LiCoO_2 (LiCoO_2 : SE layer = 92.5 : 7.5 wt%) as a positive electrode was fabricated. Neither solid electrolyte particles nor carbon conductive additives were added to the positive electrode. Fig. 4 (A) shows the initial charge-discharge curves of the all-solid-state cell In/LiCoO_2 using SE-coated LiCoO_2 and Fig. 4 (B) shows its cycle performance. The cell was charged up to 3.6 V (vs. Li-In) and then discharged to 2 V (vs. Li-In) at a constant current of 0.13 mA cm^{-2} at 25 $^\circ\text{C}$. The cell with the only SE-coated LiCoO_2 as a positive electrode as a positive electrode worked as a rechargeable battery without the addition of solid electrolyte particles and conductive additive particles to the positive electrode, and the initial discharge capacity was 45 mAh g^{-1} . The sulfide solid electrolyte layers on LiCoO_2 particles were deformed by pressing for construction of the cells. The LiCoO_2 particles partially contacted each other and electronic conduction paths are therefore formed even without conductive additives. An irreversible capacity was observed, but the reason for it has not been understood yet. One possible reason is a partial lack of lithium-ion

conduction paths to LiCoO₂, which is caused by the volume change of LiCoO₂ during charge-discharge process. In our previous works, the cell using LiCoO₂ coated with Li₃PS₄ solid electrolyte prepared from *N*-methylformamide (NMF) solution showed the capacity of 32 mAh g⁻¹ at the initial discharging process [19]. The ion conductivity of Li₃PS₄ from NMF solution (2.3×10^{-6} S cm⁻¹) was one order of magnitude lower than that of the Li₆PS₅Cl electrolyte from EtOH solution, suggesting that the cells using LiCoO₂ particles coated with electrolyte layer with higher ionic conductivity showed higher rechargeable capacities. The cycle performance showed that the cell using SE-coated LiCoO₂ retained the capacity of more than 40 mAh g⁻¹ during 10 cycles.

In our previous report, the ionic conductivity of the Li₂S-P₂S₅ solid electrolyte prepared by the pulsed laser deposition (PLD) technique was more than 10⁻⁴ S cm⁻¹ [10]. The electrochemical performance of the all-solid-state cell using LiCoO₂ coated with the Li₂S-P₂S₅ electrolyte prepared by PLD was better than the cell with LiCoO₂ coated with the Li₆PS₅Cl electrolyte from EtOH solution at the present stage. The coating of electrolytes on active material particles *via* liquid-phase process is more cost-effective than that *via* gas-phase process such as PLD, and thus conductivity enhancement of electrolyte coatings from liquid-phases is important for realizing all-solid-state batteries. Future studies on electrolyte compositions and suitable solvents are needed to prepare

solid electrolytes with higher lithium-ion conductivity.

4. Conclusions

The $\text{Li}_6\text{PS}_5\text{Cl}$ solid electrolyte was successfully prepared by dissolution-reprecipitation process *via* ethanol solution. A homogeneous solution was obtained by dissolving $\text{Li}_6\text{PS}_5\text{Cl}$ in ethanol. Crystalline $\text{Li}_6\text{PS}_5\text{Cl}$ was obtained after drying the solution. The $\text{Li}_6\text{PS}_5\text{Cl}$ electrolyte obtained from ethanol solution exhibited the conductivity of $1.4 \times 10^{-5} \text{ S cm}^{-1}$ at room temperature and the temperature dependence for conduction of 33 kJ mol^{-1} . The LiCoO_2 particles were coated with the $\text{Li}_6\text{PS}_5\text{Cl}$ electrolyte using the ethanol solution. The all-solid-state cell with the electrolyte-coated LiCoO_2 as a positive electrode operated as a rechargeable battery, and showed the higher discharge capacity of 45 mAh g^{-1} than the cell using the Li_3PS_4 -coated LiCoO_2 in our previous work. It is concluded that coating of the high ion-conducting solid electrolyte by the liquid-phase method is effective to improve the electrochemical performance of all-solid-state cells.

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Figure captions

Figure 1 (A) Raman spectra of the original $\text{Li}_6\text{PS}_5\text{Cl}$ solid electrolyte (a), the ethanol (EtOH) solution (b) and the powder prepared from the EtOH solution after drying at 80 °C under vacuum (c). The figure (B) shows X-ray diffraction patterns of the original $\text{Li}_6\text{PS}_5\text{Cl}$ solid electrolyte (a) and the powder prepared from the EtOH solution after drying at 80 °C under vacuum (b).

Figure 2 Impedance spectra of the original $\text{Li}_6\text{PS}_5\text{Cl}$ solid electrolyte (a) and the $\text{Li}_6\text{PS}_5\text{Cl}$ solid electrolyte prepared from the EtOH solution after drying at 80 °C under vacuum at room temperature (b).

Figure 3 Scanning electron microscopy and energy dispersive X-ray analysis mapping (Co, P, S and Cl) images of a LiCoO_2 particle coated with the $\text{Li}_6\text{PS}_5\text{Cl}$ solid electrolyte.

Figure 4 (A) Initial charge-discharge curves and (B) cycle performance of the all-solid-state cell In/LiCoO_2 using the LiCoO_2 particle coated with the $\text{Li}_6\text{PS}_5\text{Cl}$ solid electrolyte.

Fig. 1

Fig. 2

Fig. 3

Fig. 4