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Preparation and Microstructure of $Li_4Ti_5O_{12}$ Anode Material for Lithium-Ion Batteries

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ABSTRACT
Preparation of Spinel-$Li_4Ti_5O_{12}$ was successfully synthesized by a solid-state reaction method at 800℃, 900℃, and 1000℃. The microstructure and partical morphology behavior of $Li_4Ti_5O_{12}$ anode material was investigated by Scanning electronic microscope, X-ray diffraction. The results showed that the microstructure of $Li_4Ti_5O_{12}$ anode material of the brimmed microspheres. Heat-treated at 1000℃ most uniform structure.
Keywords – Lthium-ion batteries, $Li_4Ti_5O_{12}$, Microstructure

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I. INTRODUCTION

Recently, there has been considerable interest in $Li_4Ti_5O_{12}$ anode material for use in lithium ion batteries. Lithium ion (Li-ion) batteries have the highest performance among commercially available batteries in the respects of energy density, long life cycle, and lost costs[1]. Spinel- $Li_4Ti_5O_{12}$ has attracted attention as a promising anode material because of the no change in the unit cell volume during charge and discharge. As shown in Fig 1, $Li_4Ti_5O_{12}$ has a defect spinel framework structure with a $Fd\bar{3}m$ space group, in detail, the 32e sites are taken by oxygen atoms, 5/6 of the 16d sites are taken by Ti4+ and rest of the 16d sites are taken by Li+ [2]. Spinel-$Li_4Ti_5O_{12}$ can accommodate lithium ions during discharge, resulting in a structural transition from spinel-$Li_4Ti_5O_{12}$ to phase transformation $Li_7Ti_5O_{12}$ with out noticeable changes in the lattice parameter. During cycling processes, lithium insertion intothe spinel-$Li_4Ti_5O_{12}$ relocates lithium from of a $Li_7Ti_5O_{12}$, The electrochemical insertion of Li+ can be represented by Equation (1) as below:

$$Li_4Ti_5O_{12} + 3Li^+ + 3e^- = Li_7Ti_5O_{12} \quad (1)$$

These characteristics ensure long life cycle and very good cycle performance. The $Li_4Ti_5O_{12}$ inserts three lithium ions per formula unit, with a theoretical capacity of 175 mAhg-1, showing a voltage flat at 1.55 V a lithium electrode [3].

However, research on $Li_4Ti_5O_{12}$ mainly focuses on anode material of rechargeable lithium ion batteries using $Li_4Ti_5O_{12}$. In this paper, we preparation sample $Li_4Ti_5O_{12}$ particles via solid-state reaction and study microstructure. The microstructure and partical morphology behavior of $Li_4Ti_5O_{12}$ anode material was investigated by Scanning electronic microscope, X-ray diffraction.

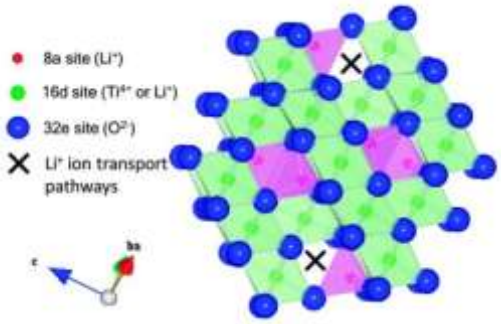

Fig.1 $Li_4Ti_5O_{12}$ crystal structure[2]

II. EXPERMENTAL

$Li_4Ti_5O_{12}$ was synthesized from TiO2 and Li2CO3 as starting materials. Stoichiometric amounts of TiO2 and Li2CO3 (Ti/Li = 5:4) were mixed in ethanol (99.9%). After ball milling for 12 h, the mixed slurry was oven-dried at 100℃. To obtain the final $Li_4Ti_5O_{12}$, the mixed precursors were heat-treated at 800℃, 900℃, and 1000℃ for 2 h under air atmosphere. The synthesized samples were characterized by An XRD experiment was conducted using a D/MAX-2500PC X-ray diffractometer. XRD spectra were recorded with Cu Kα (λ = 1.5406 A°) radiation in the diffraction angle of 2θ from 20 to 80. The morphology and microstructure were observed using a scanning electron microscopy SEM observations were conducted using a FEI Quanta 650 FEG.

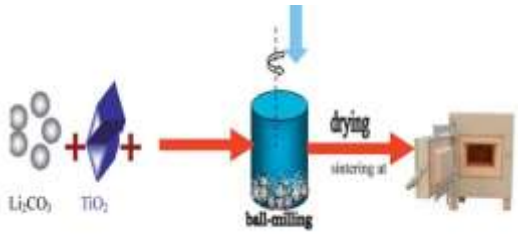

Fig.2 Schematic illustration of the preparation of the $Li_4Ti_5O_{12}$

III. RESULTS AND DISCUSSION

3.1 XRD analysis of $Li_4Ti_5O_{12}$

Fig. 3 shows the XRD patterns of the $Li_4Ti_5O_{12}$ powders. The XRD patterns show the no heat-treatment crystallization has occurred. In contrast, the patterns of the samples synthesized at 800℃, 900℃, and 1000℃ are closely in accordance with the $Li_4Ti_5O_{12}$ cubic spinel phase structure. The process exhibited diffraction peaks at 2θ (deg.) values around 37^0, 43^0, 49^0, 58^0, 63^0, 67^0 and 75^0 which corresponds to (222), (400), (331), (511), (440), (531) and (622) planes, respectively.

Because calcination temperature increases from 800-900℃ to 1000℃, the crystallinity of $Li_4Ti_5O_{12}$ improved. No obvious difference was found between the XRD patterns obtained at 800-1000℃ for 2 h. The cell parameter of the $Li_4Ti_5O_{12}$ is 83.61 nm (table 1). The XRD patterns correlate with reports from previous studies . The XRD results of the samples $Li_4Ti_5O_{12}$ obtained at 800℃, 900℃, and 1000℃ for 2 h are well defined as single-phase $Li_4Ti_5O_{12}$, with cubic symmetry, space group Fd3m, which coincides with JCPDS data, Card No. 26-1198.

Table.1 Lattice parameters of $Li_4Ti_5O_{12}$

Lattice parameters	$Li_4Ti_5O_{12}$
this work	83.61нм
Byung Gwan Lee, Jung Rag Yoon[4]	83.58нм
Xiangcheng Sun, Manu Hegde etc [5]	83.50нм

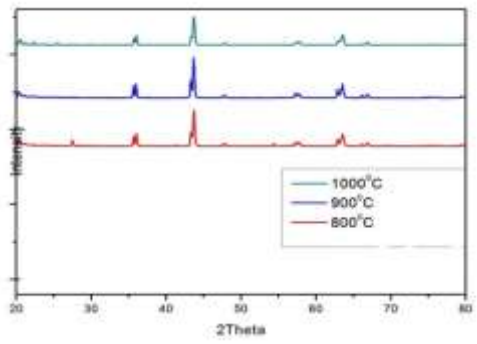

Fig. 3 XRD curves of $Li_4Ti_5O_{12}$

3.2 Microstructure of $Li_4Ti_5O_{12}$

Fig. 4 shows the SEM images of the $Li_4Ti_5O_{12}$ samples heat-treated at various temperatures for 2 h under air atmosphere. For $Li_4Ti_5O_{12}$, not heat-treated amorphous structure can be identified in the SEM image (Fig.4a). With increasing synthesis temperature, average particle diameter of the $Li_4Ti_5O_{12}$ was increased can be identified in the SEM image (Fig.4b-d). The micrographs reveal that the morphology, surface area, and particle size were affected by synthesis temperature Grain growth occurs with the size increase of crystallites in materials at high temperature. Thus, the increase of synthesis temperature results in grain growth. The microstructure of $Li_4Ti_5O_{12}$ anode material of the brimmed microspheres. Heat-treated at 1000℃ most uniform structure .

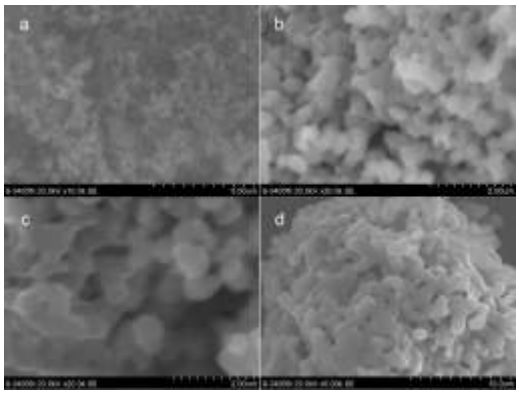

Fig. 4 SEM image $Li_4Ti_5O_{12}$ a)not heat-treatment b)800℃ c)900℃ d)1000℃

IV. CONCLUSION

In summary, pure sample taken of $Li_4Ti_5O_{12}$. The microstructure transformation behavior was investigated by XRD and SEM . The microstructure of the $Li_4Ti_5O_{12}$ anode material of the brimmed microspheres. The lattice of $Li_4Ti_5O_{12}$ is a=83.61nm.

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