Effect of Bi oxide surface treatment on 5 V spinel $LiNi_{0.5}Mn_{1.5-x}Ti_xO_4$

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Abstract

The effect of Bi surface treatment on $LiNi_{0.5}Mn_{1.5-x}Ti_xO_4$ was examined. As a result, Bi surface film around 20 nm thick was confirmed to be fabricated on the surface of 5 V spinel $LiNi_{0.5}Mn_{1.5-x}Ti_xO_4$ by transmission electron microscopy (TEM) and energy dispersion X-ray spectrometer (EDX) analysis. The Bi compound was confirmed to be Bi_2O_3 by X-ray diffraction analysis. Cycle behavior was also found to be improved by Bi treatment. A retention capacity of up to around 85% was achieved after 500 cycles at 20 °C, while a retention capacity as high as 70% was obtained after 500 cycles, even at 45 °C. Storage performance was also improved with Bi treatment. Recovery capacity of more than 90% was obtained with Bi treatment after storage for 1 week at 60 °C. The increased resistance in storage was also suppressed with Bi treatment, which would be due to the suppression of electrolyte decomposition by the Bi coating film. It was found that Bi treatment had the effect of decreasing the dissolution of Mn, Ni and Ti of the elements of the cathode active material, due to inductively coupled plasma (ICP) analysis. Based on these results, the Bi surface coating was found to have a remarkable effect on the improvement of the cells with 5 V spinel.
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1. Introduction

The use of 5 V class spinel $LiNi_{0.5}Mn_{1.5}O_4$ [3] as a cathode material is anticipated for next generation lithium ion secondary batteries. Cathode materials presently used commercially include $LiCoO_2$, $LiNiO_2$, $LiMn_2O_4$ and so on, all of which have respective merits and demerits. $LiCoO_2$ has a high capacity, but also problems of high cost and instability when in a charged state. Therefore, although it is suited for use with compact cellular phones and so on, applicability for large-sized batteries, such as those for automobile use, is problematic. $LiNiO_2$ meanwhile, has a higher capacity than $LiCoO_2$, but retains the problem of lower discharge voltage than $LiCoO_2$, and the crystal instability in a charged state. $LiMn_2O_4$ has the merits of low cost and a higher crystal stability in a charged state, making it suitable for large-sized uses such as automobile use. However, due to its lower capacity than $LiCoO_2$, it is unsuitable for portable applications. 5 V spinel, which involves substituting the portion of Mn in $LiMn_2O_4$ with Ni, Fe, Co, Cu or Cr, has a high charge-discharge potential of around 5 V versus Li.

$LiNi_{0.5}Mn_{1.5}O_4$ has a high energy density in 5 V spinel and a higher energy density than $LiMn_2O_4$. $LiNi_{0.5}Mn_{1.5}O_4$ also has a low material cost and high crystal stability when charging. The energy density is nearly equal to $LiCoO_2$. For these reasons, it has potential for use in both compact media, such as cellular phones, and also larger-sized units, such as automobiles. The 5 V spinel has been studied by many researchers. Zhong et al. [3] showed that $LiNi_{0.5}Mn_{1.5}O_4$ had a considerable rechargeable capacity in charging to 4.9 V. $LiCr_xMn_{2-x}O_4$ [4], while $LiCu_xMn_{2-x}O_4$ [5], $LiFe_xMn_{2-x}O_4$ and so on have also been reported. However, because the 5 V class spinel has a higher potential than conventional active materials, there are problems of electrolyte decomposition. Some researchers have reported on examinations of long-term reliability. Ariyoshi et al. [6] reported that positive cycle behavior was obtained by the use of a $Li[Li_{1/3}Ti_{2/3}]O_4$ anode. However, the long-term reliability was insufficient for commercial use.

To improve the lifespan, many researchers examined the element substitution of $LiMn_2O_4$. The purpose of these substitutions was to limit the dissolution of Mn from $LiMn_2O_4$ and the stabilization of the crystal structure. Recently, the substitutions of elements of $LiNi_{0.5}Mn_{1.5}O_4$ have been studied, such as $LiMg_dNi_{0.5-d}Mn_{1.5}O_4$ [7], $LiNi_{0.5}Mn_{1.5-x}Ti_xO_4$ [1,8], $LiNi_{0.5+x}Mn_{1.5-2x}Mo_xO_4$ [9]. In previous studies [1,2], the

authors studied the substitution of Mn of $LiNi_{0.5}Mn_{1.5}O_4$ for Ti. In terms of results, the cycle behavior was found to have improved due to the Ti substitution and capacity retention of around 90% after 500 cycles at 20 °C was achieved. However capacity fading at elevated temperatures, such as those above 45 °C, was still a problem, meaning further improvement was needed. Recently, the behaviors of 5 V spinel at elevated temperature were investigated [10]. The capacity fading would be due to the decomposition of the electrolyte. Because electrolyte decomposition is considered to occur at the interface of the active material and the electrolyte, control of the active material interface is an important issue. In previous studies, surface treatments have been analyzed in order to improve the long-term reliability of the cell with $LiMn_2O_4$, $LiCoO_2$ and so on. For example Al [12–14], Zr [15,16] and others [17–20] were studied. Bi-treated $LiMn_2O_4$ was reported to have a significant impact on improving lifespan in the previous study [21,22]. In this report, the effect of the Bi surface treatment on $LiNi_{0.5}Mn_{1.5-x}Ti_xO_4$ was examined.

2. Experimental

2.1. Synthesis

Li_2CO_3 was used for the Li source, while Mn–Ni–Ti complex oxide, which was gotten by firing of Ni, Mn and Ti source, was used for the precursor of $LiNi_{0.5}Mn_{1.5-x}Ti_xO_4$. The source materials were mixed and fired at 500–800 °C and 5 V spinel $LiNi_{0.5}Mn_{1.5-x}Ti_xO_4$ were obtained. $Bi(OH)_3$ was used for Bi source. $Bi(OH)_3$ and active materials were mixed by a mixing/granulating machine (SPARTAN RYUZER, Fuji Paudal Co. Ltd.) and fired at 600 °C, thus, Bi-treated 5 V spinel were obtained. The amount of Bi addition was controlled by the weight ratio of $Bi(OH)_3$ to 5 V spinel. Samples were prepared, to which 1, 2 and 5% of $Bi(OH)_3$ by weight were added. $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$ and $LiNi_{0.5}Mn_{1.41}Ti_{0.09}O_4$ were used as cathode active materials.

2.2. Analysis of active materials

The crystal structure of 5 V spinel was investigated by X-ray diffraction (XRD, Rigaku RINT-2000) analysis using a Cu Kα X-ray source. Rietveld refinement was performed using RIETAN2000 [23] to analyze the lattice constant. To investigate the Bi disperse condition on the surface of the active material, a cross-section of active material particles were analyzed by a transmission electric microscope (TEM, Hitachi, H-9000UHR II) and an Energy Dispersive X-ray spectrometer (EDX, FE-STEM Hitachi, HF-2210, EDX:NORAN VOYAGER).

2.3. Charge–discharge properties evaluations

Electrochemical properties were measured with a coin type cell. Active materials and conductive agents were dispersed into the slurry, which consisted of N-methyl pyrrolidone (NMP) with polyvinylidene fluoride (PVDF). Cathode film was fabricated on the Al current collector and the cathode was punched out with a 12 mm diameter. Li metal was used as a counter electrode. The

electrolyte composition was 1 M $LiPF_6$ in a mixture of ethylene carbonate (EC) and diethyl carbonate (DEC) in a volume ratio of EC/DEC = 30/70. The charge and discharge tests were examined with a constant current of 0.1 C, within the range 4.9 V and 3 V at 20 °C.

2.4. Cycle behavior tests

Cycle behaviors were measured with 18650 type cylindrical cells. The cathode was made by the same method with a coin type cell. Amorphous carbon was used as an anode active material, while active material and conductive agents were dispersed into the slurry with NMP and PVDF. Anode film was fabricated on the Cu current collector. The electrolyte composition was 1 M $LiPF_6$, in a mixture of EC and dimethyl carbonate (DMC) in a volume ratio of EC/DMC = 40/60. During the charge-discharge cycle tests, the charging was a constant current (CC) of 1 C (1 A) below 4.75 V, and a constant voltage at 4.75 V, until the total charging time had reached 150 min. Discharge was performed with a constant current of 1 C (1 A) until 2.5 V. Cycle tests were measured at 20 °C, 45 °C and 60 °C, respectively.

2.5. Storage tests

Storage tests were measured with cylindrical cells. The electrolyte composition was 1 M $LiPF_6$, in a mixture of EC and dimethyl carbonate (DMC) in a volume ratio of EC/DMC = 40/60 or 0.9 M $LiPF_6$, in a mixture of volume ratio of EC/DMC = 50/50. Before storage, the capacity of the cells was evaluated. During capacity measurement, charging was carried out with a constant current and voltage up to 4.75 V, and discharging was carried out with a constant current of 0.2 C until 2.5 V at 20 °C. After the cells were charged once again, cells were stored at 60 °C for 7 days. After storage, discharging was performed at 20 °C and the retention capacity was measured. Subsequently, charging and discharging were performed to measure the recovery capacity. The same storage tests were repeated moreover for 7 and 14 days, respectively, and recovery capacities of 14 and 28 days storage respectively were measured. The impedance of the cells was measured before and after storage, within the frequency range 100 kHz to 100 mHz, with amplitude of 5 mV.

2.6. Investigation of dissolution of elements

To analyze the dissolution of elements of 5 V spinel after storage, inductively coupled plasma (ICP) spectroscopy was analyzed. Measured samples included the anodes of cells after storage for 1 week at 60 °C with Bi 2 wt% treated $LiNi_{0.5}Mn_{1.41}Ti_{0.09}O_4$ and those without Bi treatment.

3. Results and discussion

3.1. Crystal structure

Figs. 1 and 2 show the XRD patterns of Bi-treated and non-treated $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$. Bi_2O_3 was detected in the active
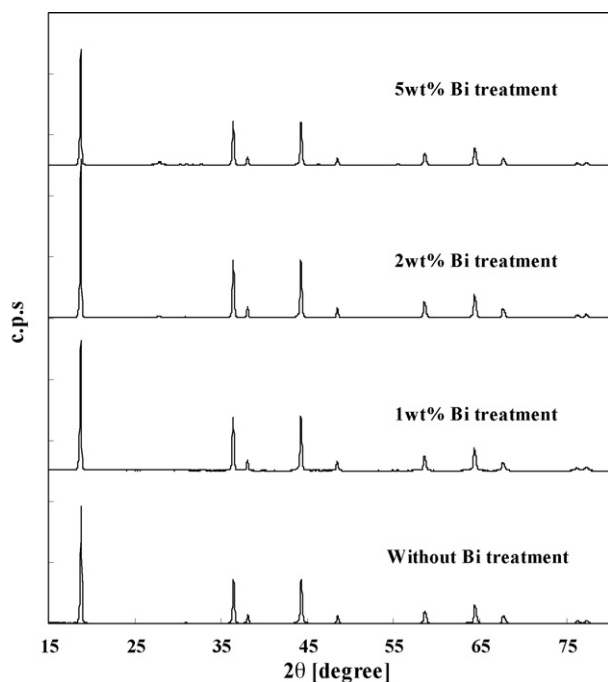
Fig. 1. XRD patterns of Bi-treated $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$.

Fig. 3. Lattice constant of Bi-treated $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$.

Bi does not diffuse into spinel. Therefore, the slight change of Mn composition of spinel due to the diffusion of Mn in spinel into a Bi compound, or the change in the oxygen content of spinel can be considered as possible explanations of the decrease of lattice parameters.

3.2. TEM observation and EDX analysis of the particle cross-section

The cross-section of active material particle was observed by TEM analysis in order to confirm the nature of the condition in which treated Bi existed in the active material particle surface. The evaluated sample was 1 wt% Bi-treated $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$. Fig. 4 shows the TEM image of Bi-treated 5 V class spinel. A film around 20 nm thick was found on the surface of the active materials. Fig. 5 shows the atomic mapping images of Bi based on EDX analysis. The surface film was found to consist of Bi. From XRD analysis, Bi_2O_3 was confirmed to exist in the prepared material and consequently, the surface film was considered to be Bi_2O_3. A similar film was found at about 20% of the whole surface in 1 wt% Bi-treated $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$.

materials. The small peak of $2\theta = 30°$ was possibly thought to be Bi–Mn complex oxide and the crystal structure remained unchanged following the Bi addition. Fig. 3 shows the lattice constant of Bi-treated $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$, which were calculated by Rietveld analysis. The lattice constant of Bi-treated spinel was found to be slightly changed by Bi content. The ionic radius of Bi^{3+}, Bi^{5+}, Ti^{4+}, Mn^{4+} and Ni^{2+} are 1.16 Å, 0.86 Å, 0.75 Å, 0.68 Å and 0.84 Å, respectively. If Bi were substituted for elements of 5 V spinel, the lattice constant would increase, however it did not increase with the Bi addition, suggesting that
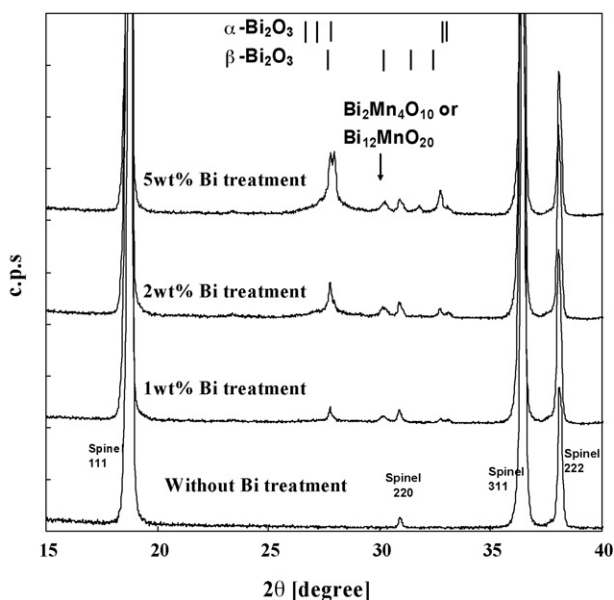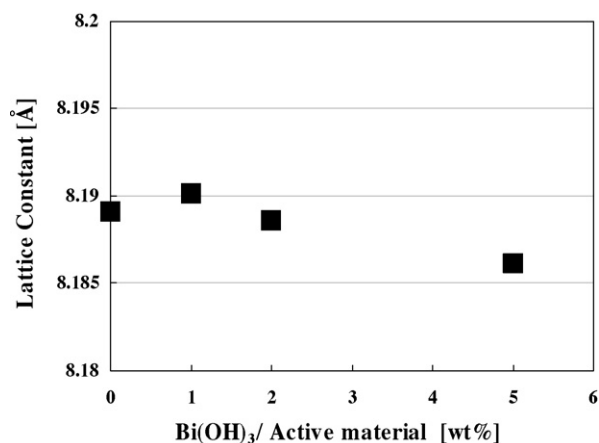

Fig. 2. XRD patterns of Bi-treated $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$ in the 2θ range from 15° to 40°.

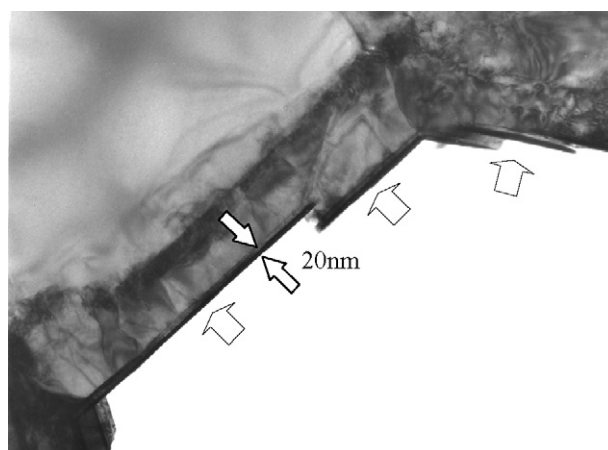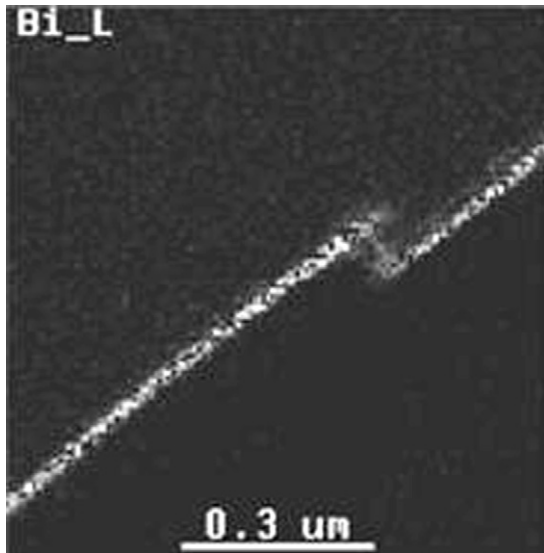Fig. 4. TEM image of the cross-section of Bi 1 wt% treated 5 V spinel.

Fig. 5. Atomic mapping image of Bi of Bi-treated $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$ by EDX analysis.

3.3. The charge–discharge characteristics of cathode active material

The charge–discharge characteristics of the cathode active materials were evaluated using a coin type cell. Fig. 6 shows the discharge curves of Bi-treated 5 V spinel. The discharge capacity decreased with the Bi amount. The discharge capacity of $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$ was around 136 mAh g^{-1}. Based on the XRD results, the Bi compound was Bi_2O_3. Bi_2O_3 is thought to be electrochemically inactive, hence the capacity is thought to decrease with an increase in the Bi amount. Fig. 7 shows the observed capacity and that calculated by assuming that the capacity of spinel remains unchanged and the Bi compound was Bi_2O_3 with density 8.9 g cm^{-3} from the literature value. The capacity of 5 V spinel with 5 wt% Bi-treated is calculated to be around 130 mAh g^{-1}, although the measured capacity was around 128 mAh g^{-1}. The potential for the composition of the cathode active material to have changed with the element diffusion from spinel to Bi oxide surface film and so on is considered,
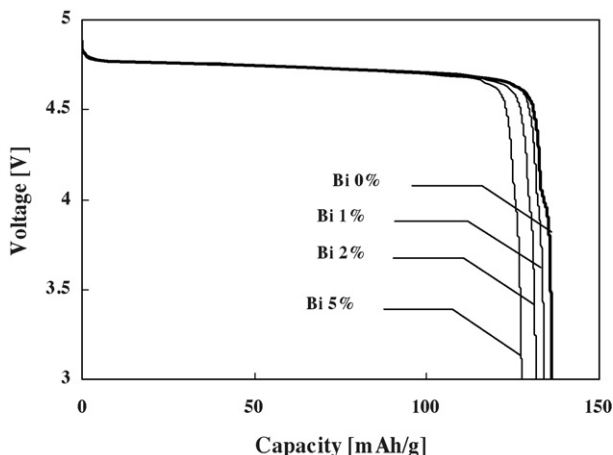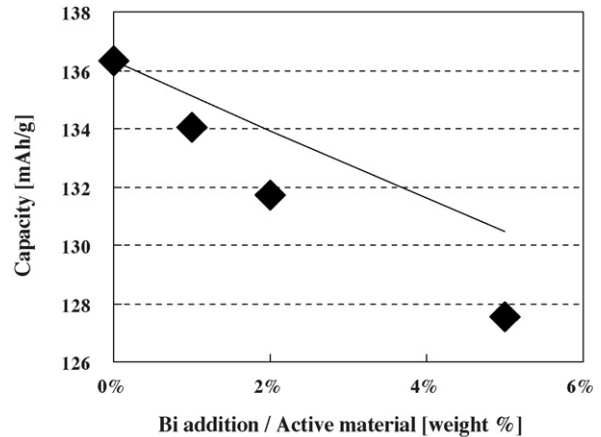

Fig. 7. Discharge capacity of the evaluation results and computation results of Bi added $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$.

although the discharge potential remains unchanged with the Bi addition. The capacity in the 4 V/Li region slightly decreased with the Bi addition. Because this capacity is considered to be due to Mn^{3+}/Mn^{4+} redox, a possible explanation for this result was a change in the oxygen deficiency of the spinel.

3.4. Cycle behaviors

Fig. 8 shows the discharge curve of the cells with $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$ cathode and amorphous carbon anode. The average discharge potential of the cells was around 4.21 V, while the 5 V region of the cathode was confirmed to be used. Because of the high potential of the anode active materials, the cells voltage was lower than the discharge potential of the cells with the 5 V spinel cathode and Li anode. The discharge curve with Bi treatment (1 wt%) was almost the same as that without Bi treatment.

Fig. 9 shows the results of cycle behaviors at 20 °C, 45 °C and 60 °C, respectively. At 20 °C, the characteristics of Bi-treated (1 wt%) and non-treated are approximately equal and capacity retention as high as 85% was achieved, retaining a high potential, even after 500 cycles. In 45 °C cycle tests, the Bi treatment was found to have a beneficial effect. Capacity retention as high

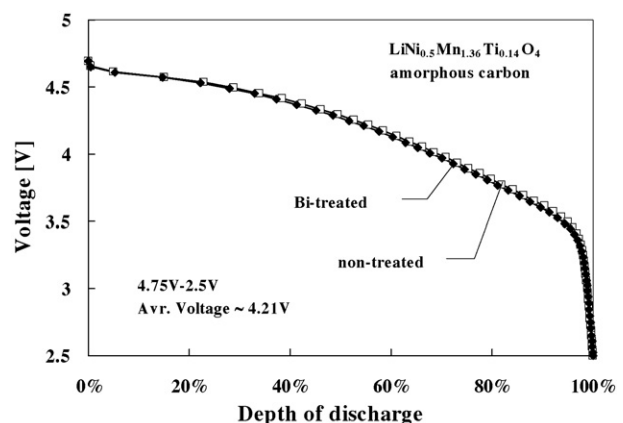Fig. 6. Discharge curves of Bi-treated $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$.

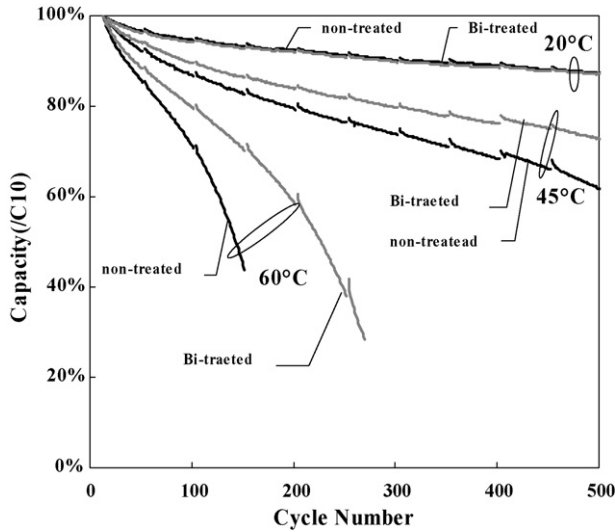Fig. 8. Discharge curve of the cells with 5 V spinel cathode and amorphous carbon anode.

Fig. 9. Cycle behavior of the cells with Bi-treated (1 wt%) and non-treated $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$ at 20 °C, 45 °C and 60 °C.

as 70% was obtained after 500 cycles at 45 °C in the case of Bi treatment. The improvement effect of the cycle characteristic was confirmed with Bi treatment also at 60 °C, however, the capacity retention after 200 cycles of Bi-treated sample was around 60%, hence there remains a problem in terms of the characteristic at an elevated temperature of 60 °C.

The capacity fading at elevated temperature can be attributed to the decomposition of electrolytes. Therefore, the suppression of the capacity fading with Bi treatment is considered to prevent this phenomenon. At 20 °C, cycle behaviors do not change by Bi treatment, which suggests that the decomposition of electrolytes was not remarkable at 20 °C. In cycle tests at 45 °C, the capacity fading ratio per cycle number with Bi treatment is smaller than that without Bi treatment, even in 500 cycles. This result suggests that the effect of Bi coat continues at 500 cycles.

3.5. Storage tests

Fig. 10 shows the recovery capacity ratio after storage for 1 week at 60 °C. In the case of $LiNi_{0.5}Mn_{1.41}Ti_{0.09}O_4$, recov-
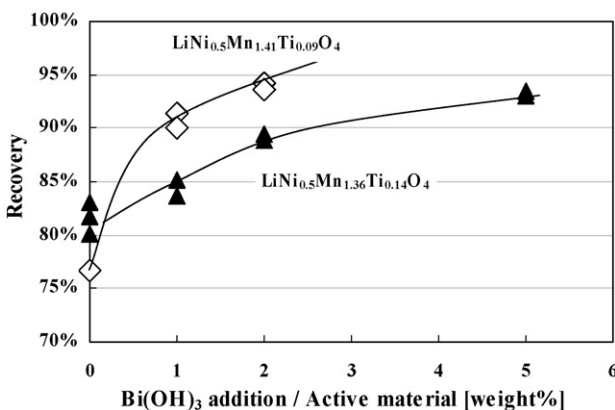

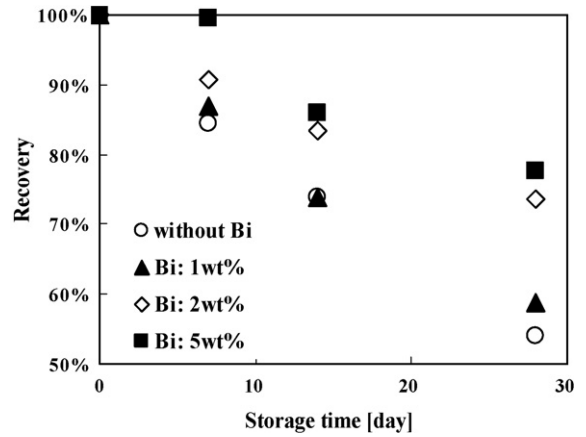Fig. 10. Recovery capacity ratio after storage for 7 days at 60 °C in case of 1 M-LiPF$_6$ EC/DMC = 40/60.

Fig. 11. Storage time dependence of recovery capacity of Bi added $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$ with 0.9 M LiPF$_6$ EC/DMC = 50/50.

ery without Bi treatment was around 78%. By the addition of 1 wt% of Bi, recovery capacity increased to more than 90%. The recovery capacity ratio with $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$, also increased with the Bi addition. The improvement effect of $LiNi_{0.5}Mn_{1.41}Ti_{0.09}O_4$ on Bi addition was higher than that of $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$, which would be due to the difference in Ti content of the cathode active material, or the difference of the grain shape and so on.

The storage performance at elevated temperature was also found to be improved by Bi treatment. These results suggest that the surface film has the effect of suppressing the capacity fading at elevated temperature. Fig. 11 shows the storage time dependence of recovery, while Figs. 12 and 13 show Cole–Cole plots of the cells without Bi treatment and that with Bi 2 wt% treated $LiNi_{0.5}Mn_{1.36}Ti_{0.14}O_4$, before and after storage at 60 °C, respectively. The resistance of the cell with Bi 2 wt% treatment was clearly smaller than that without treatment. The part with the diameter of the arc shape of Cole–Cole plots, from which the surface film component originated, was particularly small in Bi treatment. The increased resistance of the arc shape
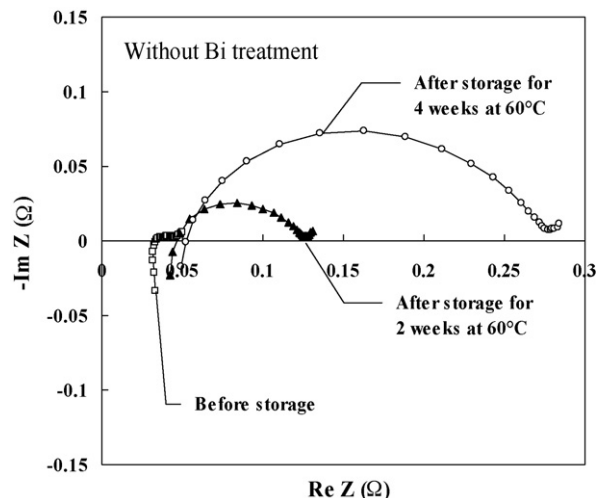

Fig. 12. Cole–Cole plots of the cell without Bi treatment, before and after storage at 60 °C.
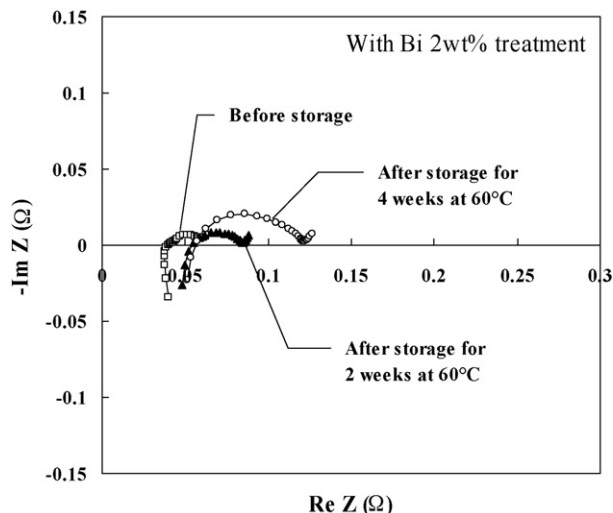
Fig. 13. Cole–Cole plots of the cell with Bi 2 wt% treatment, before and after storage at 60 °C.

was considered to be due to a double layer component, which triggers the film formation on electrode with electrolyte decomposition. Consequently, Bi_2O_3 film is considered to prevent the film formation with electrolyte decomposition.

3.6. The content of the elements on anode after storage tests by ICP

The content of the elements of the anode electrode after storage tests was investigated by ICP analysis. The element contents on anode were not perfectly consistent with the dissolution content, but were considered to be dependent on the dissolution content from the cathode active materials. The cells with 2 wt% Bi-treated $LiNi_{0.5}Mn_{1.41}Ti_{0.09}O_4$ and non-treated $LiNi_{0.5}Mn_{1.41}Ti_{0.09}O_4$ after storage for 1 week at 60 °C, were investigated. Fig. 14 shows the content of Mn, Ni, Ti and Bi on the anode after storage. The contents of Ni, Ti and Mn were
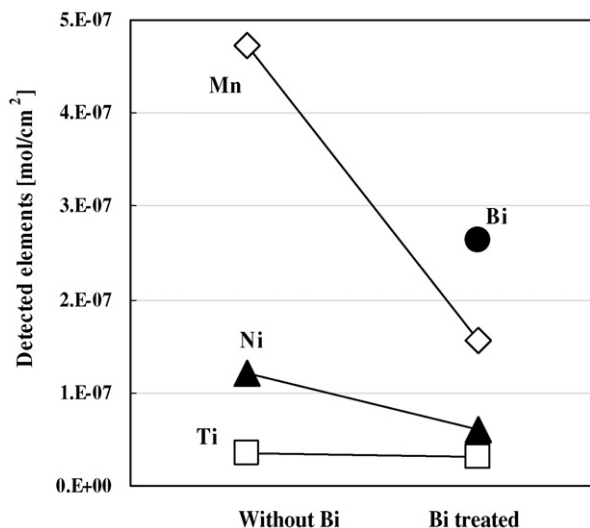

Fig. 14. The number of detected moles per unit area of Mn, Ni, Ti and Bi on the anode of the cell after storage for 1 week at 60 °C.

found to be reduced following the Bi surface treatment. The detected contents of Mn, Ni and Ti with Bi treatment was about 40% of that without Bi treatment, which suggests that the dissolution of Mn, Ni and Ti in storage was suppressed by the Bi film on the cathode active material surface. According to the storage test results, the improvement effect continued after storage for 4 weeks. This suggests that the Bi coating effect continues after 4 weeks of storage.

Electrolyte decomposition was considered as the one of the causes of the capacity fading in the 5 V spinel. On the other hand, the dissolution of the elements of active material was confirmed. As a potential cause, the decomposition of electrolyte solvents or salt causes the dissolution of elements.

4. Conclusion

The effect of the Bi surface treatment was investigated. Bi surface film was confirmed to be fabricated on the surface of 5 V spinel $LiNi_{0.5}Mn_{1.5-x}Ti_xO_4$ by TEM and EDX analysis. The thickness of the Bi film was around 20 nm, while the Bi compound was confirmed to be Bi_2O_3 by XRD analysis. The cycle behavior was found to be improved by Bi treatment. A retention capacity of around 85% was achieved after 500 cycles at 20 °C, while a retention capacity as high as 70% was obtained after 500 cycles, even at 45 °C. The improvement effect was higher at an elevated temperature, while the storage performance also improved with Bi treatment. The capacity fading after storage at 60 °C was suppressed. More than 90% of the recovery capacity after storage for 1 week at 60 °C was obtained with Bi treatment. The increase in the resistance in storage was also suppressed with Bi treatment, which would be attributable to the suppression of electrolyte decomposition by Bi film. It was found that the Bi treatment had the effect of decreasing the dissolution of Mn, Ni and Ti of the elements of cathode active material by ICP. The content of the cathode active material elements on the anode after storage for 1 week at 60 °C, decreased by about 40% in comparison with non Bi-treated. Based on the above results, the Bi surface coating was found to have a remarkable effect on the improvement of the cells with 5 V spinel.

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