The lithium-ion insertion of bulk TiO$_2$ materials [rutile, anatase, and TiO$_2$(B)] was limited to 0.5 mol in Li$_2$TiO$_3$, but nanostructured counterparts were proven to be approximately $x > 0.9$. $^{1,5}$ Due to this nanoscaled merit, many studies have been devoted to synthetic methods for maximizing its capacity as well as its rate capability. To improve the capacity and rate capability of these materials, various nanostructures [rutile, anatase, and TiO$_2$(B)] such as nanoparticle, nanorod, nanotubes, and mesoporous particles have been studied. $^{6-14}$ The achievement of high power density (rate capability) of TiO$_2$ is hindered by kinetic problems due to the slow ionic and electronic diffusion rate of the Li ion into the TiO$_2$ lattice. In this regard, the introduction of nanoparticles and higher amounts of carbon conductive agents are required to overcome the poor diffusion rate in TiO$_2$. $^{5,16}$

Nevertheless, the poor rate capability owing to a low lithium ion and electronic conductivity is still intrinsically the main obstacle for their practical applications. Lithium-ion diffusivity of TiO$_2$ is $\sim 10^{-15}$ to $10^{-13}$ cm$^2$/s, while its electrical conductivity ranges from $10^{-6}$ to $10^{-3}$ S/cm. $^{17,18}$ Accordingly, the rate performance enhancement of such TiO$_2$ is still limited because an availability of the electronic conducting phase and a percolation of the electrolyte become essentially insufficient for the higher rate capability. As a result, the enhancement of rate capability can be anticipated in the development of the TiO$_2$ material itself with a higher Li-ion conductivity. In the viewpoint of the faster Li-ion diffusion, nanocomposites or surface coating of metallic conductors on rutile or anatase TiO$_2$ are investigated. For instance, a nanocomposite with mesoporous rutile TiO$_2$ and RuO$_2$ could improve the rate capability at a 20C rate by over 50%, compared to mesoporous TiO$_2$ with a reversible capacity of $\sim 100$ mAh/g. $^{20}$ Also, carbon nanotube, anatase TiO$_2$ composite, and Ag or Sn coatings on anatase TiO$_2$ nanotubes have been reported to improve the rate capability. $^{21-24}$

Various nanostructured TiO$_2$ materials with a higher surface area and higher electrolyte-electrode contact can also be introduced for more facile Li-ion intercalation. In terms of volumetric energy density of the electrode, the best candidates among the nanostructured morphologies, such as tubes, wires, rods, or particles can be nanorod or nanoparticles. For instance, electrode density of the anatase TiO$_2$ nanorod is $\sim 1.7$ g/cm$^3$, while the TiO$_2$ nanotube is $\sim 1.3$ g/cm$^3$. $^{25}$ Unfortunately, however, anatase TiO$_2$ nanorods showed a rapid capacity fade at higher C rates than the nanowires and nanotubes even though their initial capacity is relatively higher. $^{26,27}$ For instance, the rate capacity of the anatase TiO$_2$ nanorods was improved by 62%, compared to nanorods at a 10C rate. $^{28}$ In spite of a high electrode density, the lower rate capability of the nanorod can be inferred to be originated from the relatively longer Li-ion diffusion path with respect to the nanotube. Here, it is necessary to increase Li-ion conductivity in the TiO$_2$ nanorod.

To improve both rate capability and electrode density simultaneously, another lithium intercalating compound needs to be coated on the anatase TiO$_2$ nanorod. This coating layer additionally should deliver a capacity between a voltage window of 3 and 1.5 V. In this regard, V$_2$O$_5$ is the best candidate, which shows the reversible capacity of $> 300$ mAh/g. Although its electrical conductivity of the bulk V$_2$O$_5$ is $\sim 10^{-6}$ to $10^{-8}$ S/cm, a substantial increase is expected when its dimension is in nanoscale. In this study, we report dual lithium intercalation of TiO$_2$ at V$_2$O$_5$ core-shell nanorod clusters. The nanoscale V$_2$O$_5$ coating shell layer contributes to both an additional capacity and enhanced electrical conductivity, compared to uncoated anatase TiO$_2$ nanowires. The discharge capacity of TiO$_2$ at V$_2$O$_5$ core-shell nanorods is 257 mAh/g at 0.1C rate, which is a 20% higher value than TiO$_2$ nanowires. In addition, discharge capacity of the core-shell at the 20C rate is improved by 57%, compared to TiO$_2$ nanowires.

**Experimental**

_Synthesis._—The TiO$_2$ source used for preparing the nanowires was a commercially available rutile TiO$_2$ powder (its particle size was 0.3 μm, and the particles had irregular rock-shaped morphology). $^{24,25}$ A mixture of 4 g of TiO$_2$ powder, 70 mL NaOH, and distilled water was treated with ultrasonication for 30 min, followed by treatment at 150°C in a Teflon-lined autoclave for 48 h. After treatment, the sample was treated with a 0.15 M HCl solution, followed by washing and filtering for four times, and dried at 150°C for 12 h. The dried sample was annealed further at 400°C for 3 h. For preparing TiO$_2$ at V$_2$O$_5$ core-shell nanorods, 4 g of ammonium metavanadate was dissolved in 100 mL of distilled water and then 3 g of TiO$_2$ was added in this solution, and stirred for 6 h at 90°C followed by drying at 150°C. The resultant powder was softly ground with agate mortar and pestle, and then fired at 400°C for 3 h in air.

_TEM and XRD measurements._—Transmission electron microscopy (TEM) samples were prepared by the evaporation of the samples in acetone or hexane on carbon-coated copper grids. The field-emission electron microscopic was a JEOL 2010F operating at 200 kV. Powder X-ray diffraction (XRD) measurements were carried out using a Rigaku DMax2000PC with a Cu target tube.

_X-ray absorption spectra measurement._—In situ Ti and ex situ V K-edge X-ray absorption spectra were recorded on the BL7C1 beam line of the Pohang Light Source (PLS) with a ring current of 130–185 mA at 2.5 GeV. The Si(111) double crystal monochromator has been employed to monochromatize the X-ray photon energy. In the incident V K-edge X-ray absorption fine structure (XAFS) data were collected with N$_2$ gas-filled ionization chambers for the incident and transmitted X-ray photon fluxes, while the ex situ V
K-edge XAFS was collected with fluorescence mode because of the low concentration. Energy calibration has been simultaneously carried out for each measurement with reference Ti and V metal foil placed in front of the third ion chamber. The data reduction of the experimental spectra was performed by the standard procedure reported previously.21

**Cell test.**—To test the cycle-life performance of each cathode material, a slurry was prepared by mixing the active material, Super P carbon black, and a poly(vinylidene fluoride) binder with weight ratios of 90:10:10 in n-methyl-2-pyrrolidene. The amount of active materials was ~17 mg in the composite anode. A coin-type half-cell (2016 size) contained a test electrode, a lithium-metal counter and reference electrodes, a 15 μm thick microporous polyethylene separator, and an electrolyte solution of 1 M LiPF6 in ethylene carbonate/dimethyl carbonate (1:1 vol%) (LG Chem, Korea). Cell tests were carried out at room temperature.

**Results and Discussion**

Figure 1 shows scanning electron microscope (SEM) and TEM images of the as-prepared TiO2 nanowire clusters. The SEM images present a skein of TiO2 nanowire. The TEM images show the wire diameter is ~6.5 nm and the high resolution TEM image exhibits the lattice fringes of d-spacing value of 1.9 Å, corresponding to a (200) plane of anatase TiO2, which is polycrystalline. Figure 2 shows SEM and TEM images of the TiO2 nanowire after nanoscaled V2O5 surface coating and annealing at 400°C. The initial nanowire morphology of TiO2 completely disappeared and instead, nanorod-type morphology with ~100 nm length and ~13 nm diameter are dominant, indicating that the annealing for V2O5 coating on TiO2 nanowires at 400°C results in the fragmentation of the original TiO2 nanowires. However, its clustered morphology is retained before and after annealing at 400°C, in addition to those of reference electrodes, a 15 μm thick microporous polyethylene separator, and an electrolyte solution of 1 M LiPF6 in ethylene carbonate/dimethyl carbonate (1:1 vol%) (LG Chem, Korea). Cell tests were carried out at room temperature.

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The more distinct effect of V$_2$O$_5$-surface coating onto the nanorods can be shown in the rate capability, reflecting a higher Li-ion diffusion rate, shown in Fig. 4c. Upon increasing the rate capability from 0.5, 2, 6, 10, and 20C rates, charge capacities of the V$_2$O$_5$-coated TiO$_2$ nanorods are 224, 219, 201, 183, and 173 mAh/g, respectively. A bare TiO$_2$ nanorod in an earlier paper exhibited 120 mAh/g at 10C rates. The pure TiO$_2$ nanowires show 189, 164, 137, 122, and 99 mAh/g, respectively. The electrode densities of the core-shell and TiO$_2$ nanowires were estimated to 1.9 and 1.3 g/cm$^3$, respectively (under pore volume of ~23% in the composite electrode). In consequence, volumetric capacity of the V$_2$O$_5$-coated TiO$_2$ nanorods at the 20C rate is 329 mAh/cm$^3$, while the TiO$_2$ nanowire is 128 mAh/cm$^3$. This result shows an almost 2 times higher volumetric capacity of the core-shell than the nanowires. This surface enhanced electrochemical property in the kinetics indicates that uniform formation of a very thin V$_2$O$_5$ shell layer brings a significant improvement of the Li-ion conductivity into the TiO$_2$ lattice.

In the V$_2$O$_5$-coated TiO$_2$ nanorods, therefore, it can be thought that the ability of concurrent structural transformation of the nanoscale V$_2$O$_5$ layer is important in the Li-ion insertion process. The transformation in the bulk system requires a lattice distortion in all directions to accommodate the ions, which can cause significant strain energy. In the nanosized thin layer, however, the activation barrier for this distortion is believed to be significantly decreased compared to the bulk, making it easier to relax the strain, resulting in a faster Li-ion diffusion. Namely, the nanoscaled phase is relatively favorable to surface distortion with respect to the bulk phase.

During the first Li-ion insertion, the V$_2$O$_5$ coating layer induces a drastic phase transformation: Various structural phase transformations of Li$_x$V$_2$O$_5$ have been observed depending on the lithium content. The $\alpha$- and $\varepsilon$-phases are observed for $x < 0.1$ and $0.35 < x < 0.7$, accompanied by an orthorhombic phase with increased puckering in the layers. At $x = 1$, the $\delta$-phase results from one layer gliding out of two $\alpha$- and $\varepsilon$-phases. At $x > 1$, the irreversible $\gamma$-phase is formed. Increasing the $x$ value to 3 causes the formation of the rocksalt $\omega$-phase, but a large fraction of the lithium cannot be removed upon delithiation from the $\omega$-Li$_x$V$_2$O$_5$ phase, causing a decrease in the capacity. Nanosized V$_2$O$_5$, with a particle size of ~100 nm showed a very small coulombic efficiency ratio of 76% (Fig. 5). Considering that V$_2$O$_5$-coated TiO$_2$ nanorods have a coulombic efficiency ratio of 90%, it can be estimated that the coulombic efficiency from the V$_2$O$_5$ nanoshell corresponds to 97.4% (0.86 × 65 wt % TiO$_2$ + 0.14 × 35 wt % V$_2$O$_5$) (here, 86% is the coulombic efficiency of TiO$_2$ nanorods). This indicates that the nanoscale V$_2$O$_5$ layer demonstrates quite a reversible phase transition of the $\omega$-Li$_x$V$_2$O$_5$ phase in contrast to a bulk counterpart. In the vanadium oxide bulk material, the homogenization of lithium throughout the lattice is slower, with the lithium accumulating at the core first and then additional lithium diffusing into the interior core TiO$_2$ phase. The thicker layer shows that additional lithium could not diffuse past $\omega$-Li$_x$V$_2$O$_5$ into the interior, suggesting an existence of a diffusion barrier. For the nanostructured phases, the short lithium diffusion barrier allows more Li ion to diffuse into the interior phase after the Li-rich $\omega$-Li$_x$V$_2$O$_5$ phase formed and vice versa to form a Li-deficient $\omega$-Li$_x$V$_2$O$_5$ phase upon full delithiation.

Figure 4a shows the discharge capacities vs cycle number of the V$_2$O$_5$-coated TiO$_2$ nanorods, TiO$_2$ nanowires, and V$_2$O$_5$ nanoparticles with an average size of 100 nm and carbon-coated V$_2$O$_5$ nanoparticles with an average size of 50 nm prepared by a single stage reaction of VO(OCH$_3$)$_2$ at 700°C in closed vessels in coin-type half-cells. Voltage windows for the references are between 3.5 and 1 V, and the C rate was 0.5C, except for the carbon-coated V$_2$O$_5$ nanoparticles between 4 and 2 V and at a rate of 0.1C. The V$_2$O$_5$-coated TiO$_2$ nanorods exhibit higher capacities and a higher capacity retention than TiO$_2$ nanowires and V$_2$O$_5$ references (V$_2$O$_5$ nanoparticles with a size of 100 nm and carbon-coated
In the 100 nm sized V2O5 the sample shows a slow capacity fade due to a lattice distortion induced by the volume changes. Figure 6b exhibits the rate capability of the V2O5-coated TiO2 nanorods, in addition to those of the TiO2 nanowires and nanostructured V2O5 references. Direct deposition of V2O5 gels on the nanoporous carbon prepared by the immersion of porous carbons in V2O5 hydrosol shows the highest reversible capacity of ~250 and ~210 mAh/g at 2 and 10C rates, respectively. Mesoporous V2O5 prepared by VOSO4·3H2O dissolved in water and ethanol using a block polyalkylene oxide polymer P123 surfactant displaying a pore volume of 0.56 cm3/g exhibited the highest capacity of 210 mAh/g at a 10C rate among the samples. However, this porous V2O5 is too bulky and is expected to have a low volumetric capacity. The V2O5 and carbon nanotube composite (average particle size of V2O5 was 30–40 nm) prepared by the incipient wetness impregnation of ammonium metavanadate dissolved in oxalic acid solution into carbon nanotubes exhibits a somewhat rapid capacity decrease at higher C rates. For instance, the discharge capacity of 240 mAh/g at a 2C rate decreases to 160 mAh/g at a 16C rate.

Although the V2O5 materials above generally show capacity fading, that is not a critical problem in the present study. It is because the purpose of the V2O5 nanocoating is to enhance the Li-ion conduction in the TiO2 phase. As a result, the nanoscale coating V2O5 onto the TiO2 nanorod successfully shows a better rate capability, in spite of capacity fading of the coating on the V2O5 phase. The Li-ion conduction from the Li-ion electrolyte to the TiO2 electrode has been highly enhanced through the nonstoichiometric Li12V2O7 phase in the interface region of TiO2–V2O5 core-shell, rather than in the case of direct conduction into the bare TiO2 electrode.

As shown in Fig. 7a, the intensity of the FT peaks for the V K-edge EXAFS after the first cycle abruptly increases in the V2O5-coated TiO2 nanorods, indicating the crystallinity of the surface V2O5 phase has been much more enhanced in the coated nanorods than in the pure V2O5 bulk phase (Fig. 7a3). The atomic long-range ordering in the Li12V2O7 phase can lead to an increase in the Li-ion conductivity. Figure 7b shows the corresponding Ti K-edge XANES spectra for the first cycle. In the consecutive lithiation process to the core TiO2 phase, the V2O5-coated TiO2 nanorods present a development of a well-defined anatase-like XANES peak feature for the core TiO2 phase, whereas the TiO2 nanowire shows a constant existence of highly distorted TiO6 octahedra during the first cycle. The fact means that a better Li-ion conduction by the higher crystallinity of nanoscaled V2O5 phase leads to an efficient Li-ion insertion into the core TiO2 lattice, followed by structural stability. Therefore, the V2O5-coated TiO2 nanorod results in the much easier reversible lithium–delithiation process in the coated nanorods than in the pure TiO2 nanowires. Overall, the in situ X-ray absorption spectroscopy (XAS) experiments clearly shows that there was a chemical interaction of Ti–O–V in the interface region between TiO2 and V2O5, and that the flexible structural variation in the V2O5 phase directly connected onto TiO2, which led to the easy Li-ion conduction into the TiO2 phase with respect to the bare TiO2 material during lithiation and delithiation.
Differential curves of the bulk V_2O_5 particles with a size of 300 nm during the 1st and 20th cycles are very dynamic due to structure transformation induced by Li insertion into the nanoscaled V_2O_5 phase as shown in Fig. 8. The pure V_2O_5 phase shows three well-defined voltage plateaus at 3.4, 3.2, and 2.3 V. Each one corresponds to two phase regions, \( \alpha / \epsilon, \epsilon / \delta, \) and \( \delta / \gamma \). The fourth plateau at 2 V corresponds to the formation of the \( \omega - Li_2V_2O_5 \) phase. The subsequent charge and the rest of the voltage profile do not exhibit any voltage plateau, indicating that a lithium intercalation reaction takes place in an amorphous phase, \( \omega - Li_2V_2O_5 \). The V_2O_5-coated TiO_2 nanorods show a similar differential curve, the bulk one. However, a small peak at 2 V is observed in contrast to a bulk V_2O_5 phase. This means very small-sized \( \omega - Li_2V_2O_5 \) nanocrystallites were formed. Accordingly, TEM analysis of the charged cell after the 20C rate cycling was performed (Fig. 9), and the core-shell nanorods only show \( \omega - Li_2V_2O_5 \) (Fig. 9a and b). The lattice spacing of 2.04 Á matches that of the (200) plane of cubic \( \omega - Li_2V_2O_5 \). In the case of the bulk sample after 20 cycles (Fig. 9c), most parts consist of an amorphous phase although some parts show the presence of the \( \omega - Li_2V_2O_5 \) crystallites. This ex situ result indicates that the core-shell shows quite reversible phase changes in \( \omega - Li_2V_2O_5 \), maintaining the crystallinity in contrast to the bulk counterpart.

**Conclusion**

V_2O_5-coated TiO_2 nanorods demonstrated superior rate capability with respect to TiO_2 nanowires and nanorods. In contrast to the conventional coating materials that could not react with lithium, a very thin-film layer of the V_2O_5 shell layer provided fast Li-ion diffusion into the TiO_2 core and contributed the extra capacity. Accordingly, the core-shell shows the reversible volumetric capacity of 329 mAh/cm^3 even at the 20C rate. Nanoscale V_2O_5 coating with a thickness of \(~ 6 \) nm clearly improved not only the volumetric capacity, but also the rate capability even at a high cutoff voltage. It is believed that this active shell layer can be useful in other cathode and anode materials to enhance the performance of batteries.
the core-shell nanorods during Li-ion intercalation.

Figure 8. (Color online) Differential curves of TiO$_2$, V$_2$O$_5$, and V$_2$O$_5$-coated TiO$_2$ electrodes during the 1st and 20th cycles.

Figure 9. (Color online) [(a) and (b)] TEM images of V$_2$O$_5$-coated TiO$_2$ nanorods after cycling, (c) images of nanosized V$_2$O$_5$ particle (100 nm) after cycling (red line indicates phase border between amorphous and crystalline regions), and (d) schematic view of phase transformations of V$_2$O$_5$ shell in the core-shell nanorods during Li-ion intercalation.

Acknowledgments

This work was supported by the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science and Technology (MEST) (no. R01-2008-000-20142-0). Pohang Light Source (PLS) for the XAS measurement is greatly acknowledged. Financial support from the WCU program is also acknowledged.

Ulsan National Institute of Science & Technology assisted in meeting the publication costs of this article.

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