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Chem. Mater., **Just Accepted Manuscript** • DOI: 10.1021/acs.chemmater.5b02565 • Publication Date (Web): 01 Oct 2015

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Lithiation of rutile TiO_2 coated Si NWs observed by in-situ TEM

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ABSTRACT: Lithiation of rutile TiO_2 coated Si NWs was performed inside a transmission electron microscope. TiO_2 coatings with sufficiently large coating thickness-to-NW diameter ratio induce segmented lithiation of a Si NW that reduces radial but promotes axial Li ion migration during transformation of Si to $Li_{15}Si_4$.

Increasing demand for effective energy storage has triggered a tremendous interest in high energy-density electrodes for lithium ion batteries. Commercialized lithium ion batteries with graphite as the anode have been extensively used in mobile devices. However, a new system with higher capacity and energy density is needed for hybrid electric vehicles (HEVs), plug-in hybrid electric vehicles (PHEVs), and a high power grid system.[1] Silicon is considered promising due to its high theoretical gravimetric capacity of 4200 $mAhg^{-1}$, which greatly surpasses that of graphite (372 $mAhg^{-1}$).[2] However, the application of Si has been proved challenging due to pulverization associated with a large volumetric change of 300% during lithium insertion/extraction, leading to capacity loss.[3] To overcome this problem, various approaches such as morphology control,[4] carbon-coating,[5-8] and use of polymer/organic binders,[9-10] as well as active-inactive composites[11-16] have been studied.

Surface coating of Si nanowires (NWs) by the native silicon oxide (SiO_x) has recently been proposed by Cho[14-15] and Cui[17] and their co-workers. The SiO_x shell not only enhances mechanical stability of a Si NW, but it also prevents formation of new solid electrolyte interphase (SEI), hence improving capacity retention. Other metal oxides like ZrO_2, Al_2O_3, and TiO_2 have also been considered as coating layers for lithiated Si NWs based on their different potential ranges relative to Si and thermal stabilities under SEI formation.[11-13,16] However, increasing thickness of a coating contributes to capacity fading and decreases coulombic efficiency due to the compressive stress on the Si core, making lithiation thermodynamically limited. Nevertheless, an optimized thickness of coating of an anode may sustain a longer battery life in exchange for its initial capacity being reduced.

Despite extensive study of inert oxide layers on a Si NW, understanding of lithium ion transportation through the coated Si NW is still incomplete. In many ex-situ studies, the indirect observation and quantification of Li alloying with Si has proved difficult due to heavy incorporation of carbon needed to encapsulate the oxide coated NW for conductivity enhancement.[18] The added carbon not only obscures the migration path of lithium, since Li insertion may happen in both Si and C, but it also fails to suppress large exothermic reactions between Li and Si at high temperature.[19] In contrast, an in-situ technique, where the electrochemical device is assembled inside a transmission electron microscope (TEM), for example, offers a unique opportunity to scrutinize structural changes associated with lithium insertion into a complex nanostructured system. Here, using this technique, we demonstrate for the first time how lithium inserts into and alloys with a Si NW coated with a semi-inert TiO_2 layer of varying thickness. Hence, we examine how the TiO_2 coating affects the final morphology of the inner silicon core and the resulting dimension of the entire NW during Li intercalation by varying the coating thickness-to-NW-diameter ratio (i.e. t/D ratio).

In our experiment, a batch of n-Si NWs was coated uniformly with metal oxide via atomic layer deposition (ALD), allowing precise control over the layer thickness. A rutile polymorph of TiO_2 was selected as the coating material since, upon lithiation, it turns electrically conductive to effectively enhance the electrical conduction to Si but undergoes only a 6% lattice expansion[20], and hence effectively limits the volumetric expansion of a Si NW. A mechanical aspect of rutile TiO_2 coating on a Si NW was already tested using a standard electrochemical testing cell. Using the coated NW that has the similar TiO_2-to-Si thickness ratio and was grown under the

similar method to ours, their galvanostatic cell test clearly demonstrated the improved cyclability and coulombic efficiency via improvement in mechanical stability of the system induced by coating.[12] Figure 1a shows the Si NW coated with multi-crystalline TiO_2. All the NWs including that in Figure 1a are oriented along the [001] direction, as confirmed by selected-area electron diffraction (SAED) (Figure 1b). The

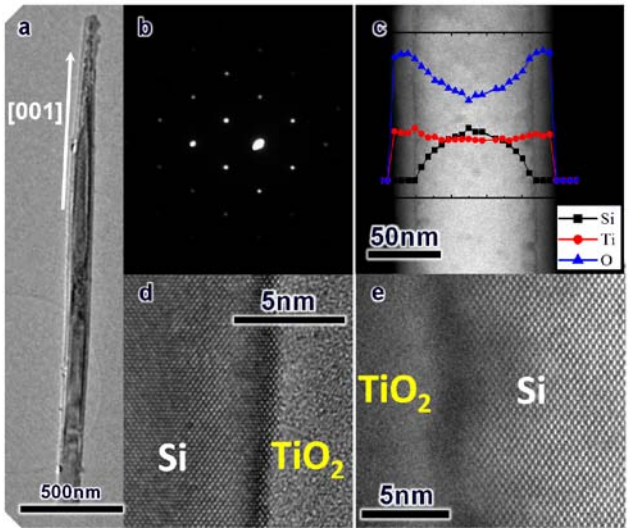

Figure 1. a. TEM micrograph of a single TiO2-coated NW, and **b.** SAED of a Si core showing [001] growth orientation, **c,d, and e.** Both EELS line scan as well as high-resolution TEM BF and STEM DF images of a Si/TiO2 interface illustrate the uniform shell thickness of TiO2 around a Si NW.

electron energy loss spectroscopy (EELS) line scan across the NW (Figure 1c) shows that the coating of TiO_2 around Si is uniform. Both bright-field (BF) TEM and dark-field (DF) STEM micrographs (Figures 1d and e) of TiO_2/Si interfaces show that the Si NW does not undergo chemical reaction with TiO_2 during coating. The coating thickness of TiO_2 is either 10 nm or 30 nm, and the NW diameter varies over the range 65-220 nm. Individual grain dimension within the TiO_2 coating varies in width but is uniform in height.

The open prototype electrochemical cell was constructed inside a TEM by loading a TiO_2-coated Si NW as one electrode and Li metal as the counter-electrode, as shown in Figures 2a and b.

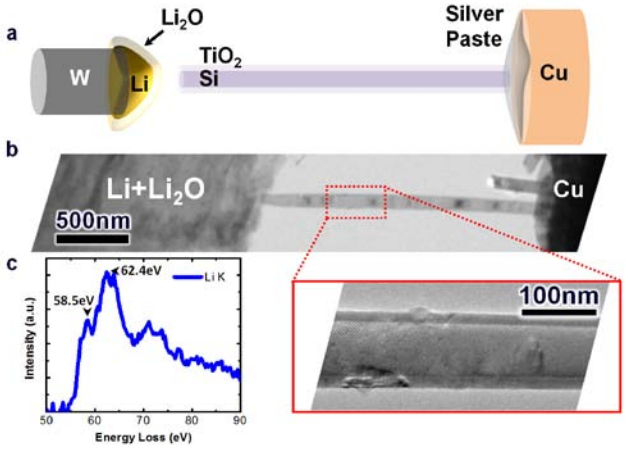

Figure 2. a. Schematic illustration of electrochemical set-up built inside TEM, **b.** TEM image of the set-up, **c.** EELS spectrum of the characteristic Li K peak from the Li_2O layer grown on Li metal.

The naturally formed Li_2O layer on the Li metal surface was considered a solid electrolyte for Li ion transport. Figure 2c shows the characteristic near-edge signal of Li K from EELS in the Li_2O layer. Charging and discharging were performed under a potentiostatic mode by applying a constant voltage across the electrodes. For the entire experiment, the potential applied to the NWs was -4V during charging and 4V during discharging, relative to the Li metal electrode. As in similar work[21], potentials larger than that of a standard electrochemical test were required to drive lithiation of Si due to the combination of low ionic conductivity of Li_2O and generally high contact resistance. Due to large contact resistance and heterogeneity between the NW and Li (held on the STM tip), any potential and current measurement during the lithiation process is not reliable, so SAED in TEM was used to confirm structural changes observed within the Si NW, especially a crystalline-to-amorphous phase transformation. The extent of lithiation was qualitatively determined by the location of the Li-NW reaction front that is driven across the entire NW.

Charging and discharging experiments of pristine phosphorous-doped Si NWs were already performed via in-situ TEM by Huang and co-workers with an electrochemical set-up similar to that used in this study.[3] Their study shows the rapid migration of Li ions along the surface of a NW, followed by diffusion into Si from the side to form an amorphous Li_xSi shell. The interfacial boundary between the amorphous Li_xSi shell and crystalline Si core moves radially inward via continuous phase transformation (e.g., $Si + Li^+ + e^- \rightarrow Li_xSi$), causing radial expansion to happen more rapidly than axial expansion. Interestingly, this radial lithiation reaches a kinetic limitation such that after a certain period of time, it no longer proceeds due to the insufficient reservoir of electrons from along the contracted Si NW core. This prevents the Si NW from reaching full lithiation.[3]

The axial migration of Li ions along the surface of a TiO_2-coated Si NW is also expected to be fast, but radial penetration of Li into the NW should be somewhat limited by the presence of the coating layer. Figures 3a, b, and c show sequential im-

ages of Li ion migration into a Si NW with TiO_2 coating (t/D=0.23) during lithiation.

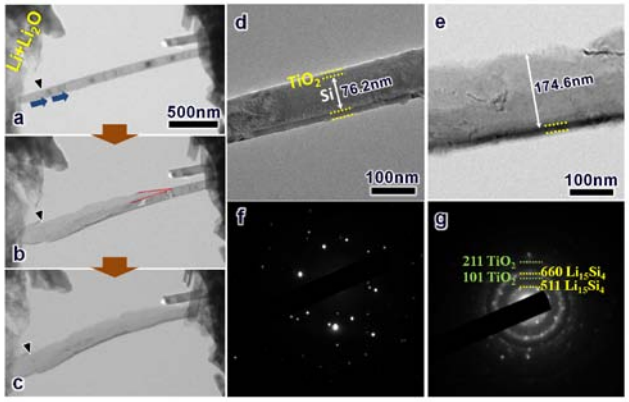

Figure 3. a, b, and c. Sequential TEM images illustrating different stages of lithiation inside a Si NW core, generating a fracture of TiO_2 shell, **d and e.** TEM images of a TiO_2-coated NW before and after lithiation, respectively and **f and g.** corresponding SAED of the these two stages.

Li ions first enter Si through a (100) facet in contact with the Li source, inducing radial expansion due to expansion along the [110] orientation.[22] This radial expansion induces the TiO_2 coating to fracture and peel off, until the entire Si core reaches full lithiation, as shown in Figures 3d and e. The change in SAED pattern upon lithiation (Figures 3f and g) provides a clear indication of the lithiated Si phase, $Li_{15}Si_4$, corresponding to full lithiation. We note that the coated NW after lithiation has undergone average radial and axial expansions of ~248% and 108%, respectively. The local radial expansion varies, however, with a maximum of only 270%, since lithiation has been limited by remnants of the TiO_2 coating. Since the coating is polycrystalline, variation in grain-to-grain bonding along the NW allows weaker bonding to generate a higher chance for cracks to initiate and propagate. Hence, the strength of bonding within TiO_2 influences the overall morphology of phases found in the lithiated Si NW. It is interesting to note that, during the lithiation of this composite NW, there is no significant change in dimension of the TiO_2 coating, implying that it functions as an effective buffer upon lithiation of Si. To prove this, an additional experiment was performed on a closed-ended NW (also capped by TiO_2) instead of an open-ended one under experimental conditions (Figure not shown). Even after a long lithiation period, no significant change was observed.

The average propagation speed of the reaction front for a pristine p-doped Si NW was found to be approximately 14 nm/s, ~10 times faster than in an undoped Si NW. This is comparable to the speed observed in an uncoated phosphorous-doped Si NW.[3] The speed of lithium migration in a coated NW, however, varies from NW to NW during lithiation due to variations in coating, i.e., the presence of differently sized and shaped grains. (See Video S1, supporting information.) Lithium ions migrating along the NW surface are hampered at certain points in time as the interfacial bonding between TiO_2 and Si is robust enough to initially constrain the diffusion of lithium. Multi-crystalline TiO_2 grains provide a very limited degree of Li ion diffusion[23] and, hence, drive Li ions inward instead, making sure that each segment of the Si NW is fully lithiated. Hence, the full lithiation can be achieved locally by continuously diverting Li ions inside Si by having a TiO_2 shell interrupting their surface migration.

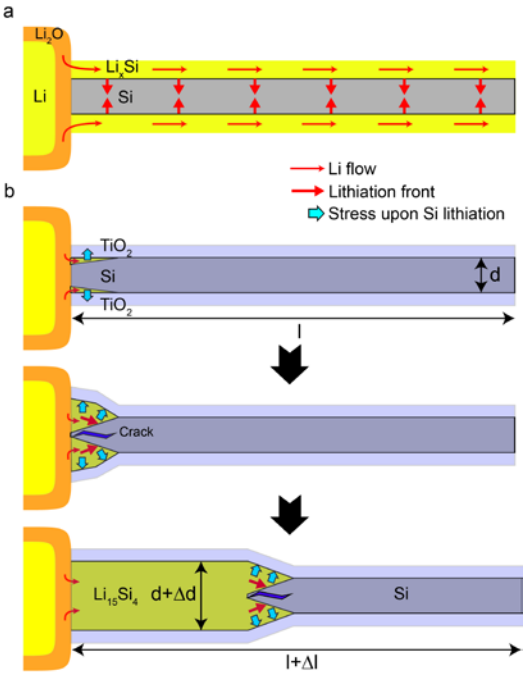

Figure 4. Schematics of lithium ion insertion **a.** into a primitive p-Si NW and **b.** into a TiO_2-coated p-Si NW.

During each segment of lithiation, the stress from a lithiated NW builds up due to continuous feeding of Li ions that induces volumetric expansion as indicated from complete amorphorization of the Si core (see schematic in Figure 4) followed by crystallization to c-$Li_{15}Si_4$, which has lower energy of formation than the amorphous phase, Li_xSi.[24]

This leads to further tearing of TiO_2 coating, allowing Li ions to initiate and repeat the lithiation process in adjacent segments of the NW until the entire coating is cracked axially. The details of this segmented lithiation are well illustrated in Figure S1. Note that the length of each segment varies according to the presumed bonding strength between TiO_2 and Si. Also note that the TiO_2 fracture parallel to the axis of lithiation indicates tensile hoop stress developed upon radial expansion of Si. A similar observation was made during in-situ TEM testing on other Si NW systems coated with polycrystalline metals including Cu[25] and Ni[26]. This fully lithiated NW, upon delithiation, performed by applying +4V relative to the Li metal, contracts both radially and axially by 21% and 3%, respectively, clearly showing some degree of integrity of the NW despite the large irreversible damage to its structure (Figure S2).

While the TiO_2 coating appears to promote lithiation of Si along the axial direction by blocking radial penetration of Li, we anticipate that such lithiation behavior should be a function of coating thickness. Here, we also tested another NW, with t/D=0.08, to observe lithiation behavior of a Si NW with thinner TiO_2 coating. Since the strength of bonding between TiO_2

grains is relatively weaker, compared to the tensile stress arising from lithiation of the Si NW core in the case with t/D=0.23, lithiation proceeds smoothly throughout the NW, with little effect of the TiO_2 coating. The final morphology after full lithiation is quite uniform throughout, similar to that of the uncoated NW, as shown

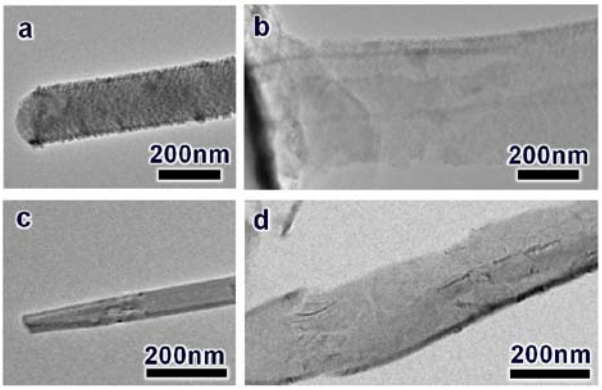

Figure 5. TEM micrographs of a Si NW with the core diameter-to-coating thickness ratio of 12:1 and 4.4:1, respectively, **a and c.** before and **b and d.** after lithiation.

in Figures 5a and b. This is very different from the NW with thicker coating that shows very rough surface profile (Figure 5c and d). This shows that an excessively thin coating has almost no mechanical influence on the NW lithiation, since the entire lithiated NW has undergone significant contraction in length (~28%) and only slightly less radial expansion than the uncoated one. The rough surface profile of the NW with thicker coating agrees well with that of the NW post-mortem examined after the standard electrochemical test that also exhibited fracture parallel to the lithiation axis.[12] The comprehensive plot in Figure 6a demonstrates the effect of the relative coating thickness (t/D) on the average radial expansion of the coated NW, based on four different NW measurements. From the plot, it is apparent that the thicker coating can suppress the NW core from expanding and somewhat maintain the integrity of a Si NW, again likely due to the stronger bonding between grains of the TiO_2 coating. In addition to the average radial expansion, average axial elongation of the coated NW upon lithiation is also plotted against t/D. A coated NW with higher t/D ratio tends to remain more elongated than one with lower ratio, due to the segmented lithiation. The overall difference in the lithiated morphology of the coated NW for both low and high t/D ratio is illustrated in Figures 6b and c and agrees well with that suggested by Cui and co-workers from their work on ex-situ electrochemical lithiation of a coated Si NW.[17] Their work, which was also performed under potentiostatic mode, involved lithiating a batch of coated Si NWs and extracting comparative statistics of these NW dimensions before and after lihiation. They concluded that coated Si NWs with lower t/D ratio exhibit both radial expansion and contraction in length upon lithiation. However, for NWs with higher t/D ratio, they observed a slight increase in both the length and diameter upon lithiation, despite that the degree of radial expansion is still more significant than that of axial expansion, in agreement with our in-situ results[17].

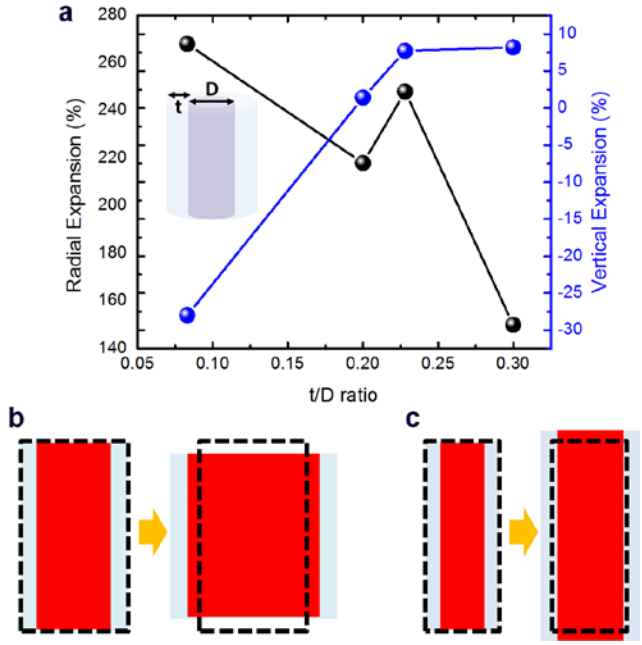

Figure 6. a. A graph showing the radial expansion and vertical expansion of a silicon core (%) against the coating thickness-to-core diameter ratio. Summarized schematics showing the final lithiated morphology of coated Si NWs with **b.** low t/D ratio vs. **c.** high t/D ratio

In summary, we have studied the nano-scale effect of TiO_2 coating on the morphological changes of a Si NW upon full lithiation by in-situ TEM. Coatings with sufficiently large t/D ratio induce segmented lithiation that promotes axial Li ion migration during transformation of Si to $Li_{15}Si_4$. Hence, the overall morphology of the coated Si NW upon full lithiation shows less radial expansion, as well as reduced axial contraction. These results confirm the effect of coating Si NWs with inert, or semi-inert oxide layers on lithiation, which may lead to a more optimized morphology control of Si nanostructures that can better accommodate anisotropy, thus improving the performance of Si as a Li-ion battery anode, while providing insight into the dynamics that occur during the lithiation process.

ASSOCIATED CONTENT

Supporting Information

Experimental details including materials, TEM specimen preparation, and characterization; Figures S1 and S2 and Video S1. This material is available free of charge via web at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest

ACKNOWLEDGMENT

This work is supported from the Center for Solar and Thermal Energy Conversion, an Energy Frontier Research Center funded

by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences under Award DE-SC0000957, and the National Science Foundation under grant DMR-0723032.

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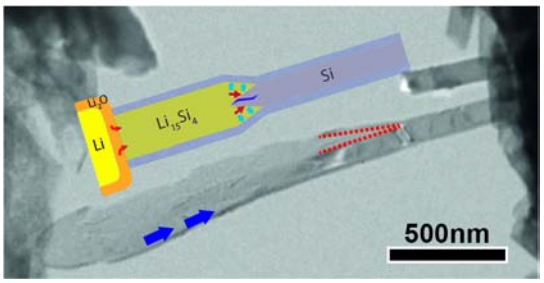
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