RAPID COMMUNICATION

Atomic layer deposition of epitaxial ZrO₂ coating on LiMn₂O₄ nanoparticles for high-rate lithium ion batteries at elevated temperature

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Abstract
Epitaxial ZrO₂ coating on LiMn₂O₄ nanoparticles using atomic layer deposition (ALD) shows high conformity, uniformity and precise thickness control at atomic scale. The thickness of ZrO₂ coating can be precisely tailored at 2 Å per ALD cycle. Cycling performance of ZrO₂-ALD-modified LiMn₂O₄ cathode material is optimized by tuning the coating thickness via varying ALD growth cycles. The optimal thickness of ZrO₂ coating for maximized electrochemical performance of LiMn₂O₄ is ~1.2 nm grown via 6 ZrO₂ ALD layers. LiMn₂O₄ nanoparticles coated with 6 ZrO₂ ALD layers deliver a very high initial discharge capacity of 136.0 mAh/g at 1C at 55 °C, significantly higher than that of bare LiMn₂O₄ (124.1 mAh/g). The effect of ZrO₂ ALD coating on improving capacity retention of LiMn₂O₄ is even more distinct at high charge/discharge rate at elevated temperature. LiMn₂O₄ nanoparticles coated with 6 ZrO₂ ALD layers after heat treatment retain a discharge capacity of 90.3 mAh/g after 100 cycles at 5C at 55 °C, while bare LiMn₂O₄ nanoparticles exhibit a final capacity of 58.8 mAh/g. ZrO₂-ALD-modified LiMn₂O₄ nanoparticles show significantly improved specific capacity and enhanced cycleability at high rate at elevated temperature due to the protective effect of ultrathin and high-quality ALD surface coatings.

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Introduction
High power lithium ion batteries have attracted tremendous attentions due to their growing applications in hybrid electric vehicles (HEVs) and full electric vehicles (EVs) [1,2]. Capacities...
and rate capabilities of lithium ion batteries can be improved by using nanostructured electrode materials. In comparison with micro-sized materials, nanomaterials provide much shorter lithium ion diffusion path and offer more active sites for lithium ion insertion and extraction due to the larger surface-to-volume ratio. Excellent cycling stability and high rate capability have been achieved for both nanostructured cathode and anode nanomaterials [3,4]. Nanosized spinel LiMn2O4 has been demonstrated as a promising cathode material for applications in high-rate lithium ion batteries due to its advantages such as high power and energy densities, low cost, environmental friendliness and safety [5-7]. However, LiMn2O4 suffers from inevitable manganese (Mn) dissolution, phase transition and electrolyte decomposition during lithiation and delithiation processes, resulting in significant capacity fading during long-term cycling, especially at elevated temperature [8-11]. Mn3+ dissolution into the electrolyte is attributed to the attack from hydrofluoric acid (HF) formed by hexafluorophosphate (LiPF6) and residual H2O in the commercial electrolyte [12]. This disproportionate degradation of LiMn2O4 is considered as the major factor leading to poor capacity retention in cycling performance. The phase and structural stability of LiMn2O4 is important for having reversible charge and discharge reactions. Phase transition of LiMn2O4 can be effectively alleviated via choosing suitable operating voltage range for lithium ion intercalations to preserve highly ordered distribution of lithium ions in LiMn2O4 structure [9]. Decomposition of organic electrolyte on the surface of cathode is induced by the high oxidation effect of Mn3+/Mn4+ redox, which considerably increases the charge transition resistance and affects the lithium ion diffusion by forming the solid electrolyte interphase (SEI) layer [10]. The three factors summarized above together attribute to the inferior cycleability of LiMn2O4 cathode material, especially at elevated temperature. Therefore, interface between LiMn2O4 particles and surrounding electrolyte plays a critical role for electron and lithium ion transfer in the cathode [13].

Artificial preparation of a protective coating on LiMn2O4 particle is an effective approach to optimize electrochemical performance of LiMn2O4 electrode [12,14,15]. Electrochemical performance of LiMn2O4 cathode can be improved by coating it with various amphoteric oxide coatings, such as Al2O3, ZnO, ZrO2, SiO2, TiO2, Cr2O3 and SnO2 [16-21]. The protective oxide film serves as an effective HF scavenger to suppress Mn dissolution. Furthermore, the oxide coating separates LiMn2O4 particles from electrolyte to alleviate decomposition of electrolyte, and acts as a solid framework to restrict the unexpected phase transition of LiMn2O4, contributing to better structural stability and cycleability [11].

Due to the large surface area and complex structure, surface coatings on nanostructured materials synthesized via traditional wet chemical methods usually lack conformality, completeness and uniformity, and the thickness of coating layer cannot be precisely controlled at the sub-nano scale. Electrochemical performance of oxide-coated electrodes can be affected by qualities of surface coatings such as adhesion between coatings and host materials, conformality, uniformity, thickness, specific surface area and crystallinity of surface coatings [22]. Atomic layer deposition (ALD) is an advanced thin film deposition technique involving a sequence of chemisorption and self-terminating surface reactions [23-25]. In a typical ALD run, the initial hydroxylation on the surface of the substrate using H2O precursor yields conformal and full coverage of the host material with hydroxyl groups, followed by purging the residual H2O and by-products with inert gas flux. Subsequently, the organometallic precursor is introduced to react with hydroxyl groups, and thus a monolayer of oxide grows on the surface of substrate bridged by oxygen bonds. After the second purging step to remove extra organometallic precursor and by-products, the continuative hydroxylation takes place to replace organic groups on the surface of organometallic precursor with hydroxyl groups for the second monolayer growth of oxide coating. As a result, sequential ALD cycles proceed step-wise in self-limiting surface reactions as mentioned above, separated by purge steps, and the oxide film grows monolayer by monolayer at atomic scale. Hence, films grown using ALD are typically uniform, dense, homogenous, pinhole-free, and extremely conformal to the underlying substrate, and the thickness of coatings can be precisely controlled at atomic level [26-31]. It is also noted that the ALD coating is chemically bonded to the substrate, while there is merely physical contact between the coating synthesized via conventional wet chemical methods and the substrate. Another distinct advantage of ALD technique is that it allows for direct low-temperature (< 200 °C) preparation of crystalline oxide coatings without post heat treatment, such as ZrO2 [32,33] and V2O5 [34].

ZrO2 coating deposited on LiMn2O4 nanoparticles via conventional sol-gel method has been demonstrated as a remarkable HF scavenger to suppress Mn dissolution and as an artificially robust interface to significantly reduce the deleterious reaction between LiMn2O4 and electrolyte [19]. Among various oxide coatings, ZrO2 has shown excellent structural and thermal stability at elevated temperature [11]. ZrO2 ALD coating for surface modification of micro-sized LiMn2O4 particles has been demonstrated to improve specific capacity and cycling stability of LiMn2O4 cathode at elevated temperature in our previous work [32]. The effect of ZrO2 ALD coating on enhancing electrochemical performance of LiMn2O4 cathode is more phenomenal at elevated temperature than at room temperature. As nanostructured battery electrodes have attracted tremendous attention due to their enhanced surface area and improved high-rate performance compared to micro-sized electrode materials, in this work we deposit ultrathin ZrO2 coating on nanosized LiMn2O4 particles via ALD to explore the interesting growth and crystalline/amorphous phases of ZrO2 ALD films and to further improve high-rate electrochemical performance of LiMn2O4 at elevated temperature. Cycling performance of ZrO2-coated LiMn2O4 nanoparticles is optimized by tuning the coating thickness at atomic scale via varying ALD growth cycles, indicating that the optimal thickness of ZrO2 coating is ~1 nm grown via 6 ZrO2 ALD layers. The ZrO2 ALD-modified cathode shows improved specific capacity and enhanced cycleability compared to bare composite electrode at high charge/discharge rate at elevated temperature, which can be considered as a promising electrode for high-power lithium ion batteries in elevated-temperature environment.

Experimental section

Atomic layer deposition of ZrO2 coating on LiMn2O4 nanoparticles

LiMn2O4 nanoparticles were purchased from MTI Corporation (marked as LMO). Atomic layer deposition of ZrO2 coating on
Atomic layer deposition of epitaxial ZrO2 coating on LiMn2O4 nanoparticles

LiMn2O4 nanoparticles was carried out in a Savannah 100 ALD system (Cambridge NanoTech Inc.) at 120 °C using Zr(OC(CH3)3)4 (Zirconium tert-butoxide, ZTB) and H2O as precursors with exposure time of 0.25 and 0.015 s, waiting time of 5 and 5 s and purge time of 60 and 40 s, respectively. The two self-terminating reactions involved in this ZrO2 ALD growth are described by the following reactions [32]:

\[
\text{Zr(OH)}^+ + \text{Zr(OC(CH3)3)4} \rightarrow \text{Zr-O-Zr(OC(CH3)3)^+} + (CH3)2COH
\]

\[
\text{Zr-O-C(CH3)3}^+ + \text{H2O} \rightarrow \text{Zr(OH)^+} + \text{CH3-C(CH3)2.}
\]

LiMn2O4 nanoparticles with n ZrO2 ALD layers were marked as n ZrO2 ALD LMO. The heat treatment of 6 ZrO2 ALD LMO was performed at 450 °C for 3 h in air (denoted as 6 ZrO2 ALD LMO HT).

Characterizations

The crystallographic structure of LiMn2O4 nanoparticles was examined by X-ray diffraction (XRD) using a Rigaku MiniFlex X-ray diffractometer with Cu Kα radiation at a scan rate of 2°/min. Surface morphology, particle size and energy dispersive spectroscopic (EDS) mappings of bare and ZrO2 ALD modified LiMn2O4 nanoparticles were observed using a FEI Quanta 3D FEG field emission scanning electron microscopy (FESEM). Transmission electron microscopy (TEM) images with EDS were captured on a FEI Tecnai G2 FEG instrument at an acceleration voltage of 300 kV, to examine morphology and structure of ZrO2 coatings. Surface compositions of LiMn2O4 nanoparticles coated with 5 ZrO2 ALD layers were analyzed via X-ray photoelectron spectroscopy (XPS) using an AXIS 165 spectrometer using a twin-anode Al Kα (1486.6 eV) X-ray source with the charge neutralization function being turned on. All the XPS spectra were calibrated according to the binding energy of the C1s peak at 284.8 eV.

Electrochemical measurements

Bare composite electrode (marked as B-E) was composed of 80% pristine LiMn2O4 nanoparticles, 10% acetylene black (conductive carbon, Alfa Aesar, 99.5%) and 10% polyvinylidene fluoride (PVDF, Alfa Aesar) as the binder. ZrO2 ALD modified composite electrodes were denoted as n ZrO2 ALD LMO-E consisting of LiMn2O4 nanoparticles coated with n ZrO2 ALD layers, conductive carbon and PVDF binder at the same weight ratio of 8:1:1. These cathodes were assembled into two-electrode CR2032-type coin cells for electrochemical measure, with metallic lithium foil as the anode, Celgard-2320 membrane as separator; electrolyte was 1 M LiPF6 dissolved in ethylene carbonate (EC) and dimethyl carbonate (DMC) and diethyl carbonate (DEC) at a volumetric ratio of 1:1:1. Galvanostatic charge and discharge were performed at different current densities in a voltage range of 3.4–4.5 V using an 8-channel battery analyzer (MTI Corporation) at elevated temperature (55 °C).

Results and discussion

Figure 1 shows the XRD pattern of bare LiMn2O4 nanoparticles. All distinct XRD peaks are indexed as the spinel cubic structure of LiMn2O4 with a Fd3m space group (JCPDS: 35-0782), indicating the high purity and crystallinity of LiMn2O4 nanoparticles.

The pristine LiMn2O4 nanoparticles have an average particle size of ~250 nm with a smooth polyhedron shape as observed in FESEM image in Figure 2a. To study details of ZrO2 ALD coating, LiMn2O4 nanoparticles are coated with thick ALD film via 50 ALD growth cycles (marked as 50 ZrO2 ALD LMO). Morphology and structure of 50 ZrO2 ALD LMO are examined under TEM as presented in Figure 2b. The shape and morphology of LiMn2O4 nanoparticles coated with 50 ZrO2 ALD layers are almost identical to those of bare LiMn2O4 particles, indicating conformality of ZrO2 ALD coating. Figure 2b shows conformal and homogeneous ZrO2 ALD coating on the surface of LiMn2O4 nanoparticles. The corresponding EDS spectrum in Figure 2c captured from the marked circle in Figure 2b confirms the existence of ZrO2 coating. The Cu peaks are from the copper grid where the TEM sample is placed. 50 ALD growth cycles contribute to a thickness of ~10 nm for ZrO2 ALD coating, corresponding to the ZrO2 ALD growth rate of ~2.0 Å/cycle. As such, the thickness of ZrO2 ALD coating on LiMn2O4 nanoparticles can be precisely tailored at atomic scale. Furthermore, in contrast to deposition of crystalline ZrO2 film on micro-sized LiMn2O4 particles, ZrO2 ALD coating on LiMn2O4 nanoparticles is partially amorphous and partially crystalline. More details for ZrO2 ALD growth on the surface of LiMn2O4 nanoparticle are obtained after obtaining HRTEM image of ZrO2 film as shown in Figure 2d. It can be seen that ZrO2 coating on LiMn2O4 nanoparticle is composed of two distinct layers in different textures and structures. The external layer of ZrO2, as shown as layer 2, is amorphous with a uniform thickness of ~2 nm, while the layer inside (layer 1) shows formation of epitaxial layer of ZrO2 following the orientation or lattice fringes of LiMn2O4 nanoparticles. These two distinct ZrO2 layers on the underlying LiMn2O4 illustrate the evolution of ZrO2 ALD coating on LiMn2O4 nanoparticles from amorphous to crystalline phase. Furthermore, Figure 3 shows the EDS mappings of Zr, Mn and O elements from agglomerated LiMn2O4 nanoparticles with 50 ZrO2 ALD coating layers, indicating the uniform
ZrO$_2$ ALD coating on every individual LiMn$_2$O$_4$ nanoparticle. In comparison with facile deposition of crystalline ZrO$_2$ film on micro-sized LiMn$_2$O$_4$ particles using the same ZrO$_2$ ALD process [32], ZrO$_2$ ALD film on nano-sized LiMn$_2$O$_4$ particles is more difficult to grow epitaxially. We speculate that factors such as morphology, particle size, surface energy and grain size of different LiMn$_2$O$_4$ particles will influence the ZrO$_2$ ALD growth process, because ZrO$_2$ ALD growth starts from chemical bonds with the surface of LiMn$_2$O$_4$. There is ongoing work in our laboratory to study the effect of ZrO$_2$ ALD coating on various nanostructured cathode materials.

To confirm the epitaxial growth of ZrO$_2$ coating on the surface of LiMn$_2$O$_4$ nanoparticles, X-ray photoelectron spectroscopy (XPS) is further employed to analyze surface composition of LiMn$_2$O$_4$ particles coated with 50 ZrO$_2$ ALD layers. In order to minimize electronic charging effect in XPS measurement, the charge neutralization function is turned on. Figure 4 presents the Zr 3d XPS pattern captured from 50 ZrO$_2$ ALD LMO particles. Two distinct Zr peaks located at the binding energies of 182 and 168 eV are detected, which are speculated to be from the two formed layers in ZrO$_2$ coating in Figure 2d. The representative Zr 3d peak positioned at 182 eV (consistent with the standard value of ZrO$_2$ in NIST XPS database) can be attributed to the stoichiometric and amorphous external ZrO$_2$ layer (layer 2 in Figure 2d). On the other hand, the other broad peak at 168 eV that has some shift from the standard binding energy of Zr 3d in ZrO$_2$ may be from the crystalline ZrO$_2$ layer (layer 1 in Figure 2d) which may have lattice change due to possible epitaxial growth of ZrO$_2$ coating on the surface of LiMn$_2$O$_4$ particles, indicating the complex chemical bonding environment for Zr element in the ZrO$_2$ ALD coating (50 ZrO$_2$ ALD layers) on LiMn$_2$O$_4$. Due to the chemical bonding in ZrO$_2$ ALD growth on LiMn$_2$O$_4$ substrate via oxygen bonds (LiMn$_2$O$_4$–O–ZrO$_2$), the amorphous ZrO$_2$ film is apt to crystallize following the crystalline orientation or lattice fringes of LiMn$_2$O$_4$ under vacuum during ZrO$_2$ ALD growth process even at very low temperature (120 °C), and thus, this epitaxial growth yields two ZrO$_2$ layers in HRTEM observation (Figure 2d). As a result, the epitaxial ZrO$_2$ layer inside (layer 1) has very different chemical bonding environment for Zr in comparison with amorphous ZrO$_2$ in the layer outside (layer 2), leading to the shift of Zr 3d peak in XPS spectrum in Figure 4.

ZrO$_2$ coating on LiMn$_2$O$_4$-based cathode material mainly serves as an effective HF scavenger to suppress Mn dissolution and acts as an artificial interface to reduce deleterious reaction between LiMn$_2$O$_4$ and electrolyte for improved cycleability of LiMn$_2$O$_4$. Furthermore, the conformal ZrO$_2$ coating on LiMn$_2$O$_4$ particles can average distribution of electrons and lithium ions on the whole surface of LiMn$_2$O$_4$ particles; hence, electrochemical polarization from electron conglomeration and concentration gradient of Li$^+$ can be significantly reduced in the cathode, especially under

Figure 2 (a) FESEM image of bare LiMn$_2$O$_4$ nanoparticles; and (b) TEM image, (c) EDS spectrum captured from the area in the marked circle in (b) and (d) HRTEM image of LiMn$_2$O$_4$ nanoparticles coated with 50 ZrO$_2$ ALD layers.
high charge/discharge rates at elevated temperature \cite{11}. Therefore, the thickness of ZrO\textsubscript{2} film needs to be optimized to maximize the electrochemical performance of LiMn\textsubscript{2}O\textsubscript{4}. ZrO\textsubscript{2} coating with optimal thickness is supposed to be sufficiently thick to consume HF acid in electrolyte but not too thick to slow down diffusion of lithium ions at high charge/discharge rate. Our previous work has demonstrated that ultrathin ZrO\textsubscript{2} coating on micro-size LiMn\textsubscript{2}O\textsubscript{4} particles for enhancing electrochemical performance of LiMn\textsubscript{2}O\textsubscript{4} is more effective than coating on the entire composite electrode composed of LiMn\textsubscript{2}O\textsubscript{4} particles and carbon/PVDF network, and the effect of oxide ALD coatings on improving capacity retention and increasing specific capacity of LiMn\textsubscript{2}O\textsubscript{4} is more phenomenal at elevated temperature than at room temperature \cite{11,22,32}. In the present work, we evaluate the effect of ZrO\textsubscript{2} ALD coating on LiMn\textsubscript{2}O\textsubscript{4} nanoparticles for high-rate cycling performance at elevated temperature. In order to optimize electrochemical performance of ZrO\textsubscript{2}-modified LiMn\textsubscript{2}O\textsubscript{4} nanoparticles at elevated temperature (55 °C), thickness of ZrO\textsubscript{2} coating on LiMn\textsubscript{2}O\textsubscript{4} nanoparticles is finely tuned by using various ALD growth cycles, such as 2, 6 and 10 ALD growth cycles, corresponding to the thickness of 0.4, 1.2 and 2.0 nm, respectively. The ALD-coated composite electrodes are prepared by mixing these ALD-coated LMO particles with conductive carbon and PVDF binder at the same weight ratio of 8:1:1 (marked as 2, 6 or 10 ZrO\textsubscript{2} ALD LMO-E), respectively. Figure 5a and b shows the initial charge and discharge curves and corresponding cycling performance of ZrO\textsubscript{2}-modified LiMn\textsubscript{2}O\textsubscript{4} cathodes in comparison with bare composite electrode at a high current...
density of 120 mA/g (≈ 1 C) in a voltage range of 3.4-4.5 V at 55 °C. Electrochemical measurements indicate that the optimal thickness of ZrO2 coating for maximized performance of LiMn2O4 is ≈ 1.2 nm grown via 6 ZrO2 ALD layers. LiMn2O4 nanoparticles coated with 6 ZrO2 ALD layers deliver a very high initial discharge capacity of 136.0 mAh/g at 1C at 55 °C, significantly higher than that of bare LiMn2O4 (124.1 mAh/g). The improved initial capacities of 6 ZrO2 ALD LMO-E can be attributed to the effective suppression of Mn dissolution and electrolyte decomposition at elevated temperature by conformal, uniform and complete coverage of ZrO2 coating on the surface of individual LiMn2O4 particle. After 100 electrochemical cycles at elevated temperature, 6 ZrO2 ALD LMO-E retains a specific capacity of 111.3 mAh/g, corresponding to an average capacity loss of 0.25 mAh/g per cycle. The sample 10 ZrO2 ALD LMO-E only shows an initial capacity of 106. 6 mAh/g, illustrating that ZrO2 coating thicker than 2 nm can significantly affect diffusion of lithium ions. On the other hand, 2 ZrO2 ALD layers with the thickness of 0.4 nm on LiMn2O4 particles are not sufficient to completely scavenge the HF acid, resulting in the apparent capacity fading from 135.1 to 97.1 mAh/g after 100 electrochemical cycles, similar to the case of B-E. It is thus found that electrochemical performance of LiMn2O4 nanoparticles modified by ZrO2 ALD layers is very sensitive to the coating thickness. The remarkable cycling stability of 6 ZrO2 ALD LMO-E after the 50th cycle indicates that the residual ZrO2 protective film after consumption of HF acid is facile for lithium ion diffusion and can effectively alleviate decomposition of electrolyte and reduce electrochemical polarization on the surface of LiMn2O4 particles.

The effect of tuning ZrO2 ALD coating thickness on improving capacity retention of LiMn2O4 is even more phenomenal at high charge/discharge rate at elevated temperature. Figure 6a and b presents electrochemical performance of ZrO2-modified LiMn2O4 cathodes subject to a very high current density of 600 mA/g (≈ 5 C) at 55 °C. The 2 ZrO2 ALD LMO-E delivers the highest initial discharge capacity of 123.4 mAh/g, higher than 112.7 mAh/g from 6 ZrO2 ALD LMO-E and 88.5 mAh/g from 10 ZrO2 ALD LMO-E as well as 114.6 mAh/g from B-E, indicating that thickness of ZrO2 coating significantly influences diffusion of lithium ions under such a high current density. During subsequent
Electrochemical cycles at elevated temperature, B-E suffers from faster capacity fading as shown in Figure 6b due to the attack from acidic HF and decomposition of electrolyte on the surface of LiMn2O4 nanoparticles. In contrast, LiMn2O4 nanoparticles coated with 6 ZrO2 ALD layers retain a discharge capacity of 76.9 mAh/g after 100 cycles, corresponding to a capacity retention of 68.2%, while bare LiMn2O4 nanoparticles exhibit a final capacity of 58.8 mAh/g and a capacity retention of only 51.4%. 2 ZrO2 ALD LMO-E particles coated with the optimal 6 ZrO2 ALD layers deliver because 10 ZrO2 ALD layers are overly thick and slow down scavenging the acidic HF. On the other hand, 10 ZrO2 ALD layers with a thickness of 0.4 nm are not sufficient to scavenge the acidic HF. On the other hand, 10 ZrO2 ALD layers with a thickness of 0.4 nm are not sufficient to scavenge the acidic HF. Therefore, 6 ZrO2 ALD layers (as thin as 1.2 nm) demonstrate to have the optimal thickness for maximized electrochemical performance of LiMn2O4 nanoparticles for applications in high-power lithium ion batteries at elevated temperature.

Conclusions

Ultrathin and highly-conformal ZrO2 coatings have been facilely deposited on LiMn2O4 nanoparticles using atomic layer deposition. Epitaxial growth of ZrO2 on underlying LiMn2O4 nanoparticles has been observed using HRTEM and XPS measurements. Crystalline ZrO2 ALD coating can grow on the surface of LiMn2O4 nanoparticles following the crystalline orientation of LiMn2O4 in low temperature ALD process with the growth of amorphous ZrO2 ALD film, yielding two distinguishing layers in the ZrO2 coating. The thickness of ZrO2 coating can be precisely tailored at ~2 Å per ALD cycle. The optimal thickness of ZrO2 coating for maximized electrochemical performance of LiMn2O4 nanoparticles is 1.2 nm (6 ZrO2 ALD layers). LiMn2O4 nanoparticles coated with the optimal 6 ZrO2 ALD layers deliver an initial discharge capacity of 136.0 mAh/g at 1C and 112.7 mAh/g at 5C at 55 °C. Surface modification of LiMn2O4 nanoparticles with ZrO2 coating has led to the remarkably enhanced high-rate electrochemical performance of LiMn2O4 at elevated temperature.

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