Sputtering of $LiCoO_2$ Thin Films

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Lithium cobalt oxide (LCO) cathode has been widely applied in 3C products (computer, communication, and consumer), and LCO films are the most promising cathode materials for thin-film lithium batteries (TFBs) due to their high volumetric energy density and favorable durability. Magnetron sputtering deposition technology realizes the merits of high speed, low temperature, and low toxicity. The magnetron sputtering technology perfectly fits the microelectronics, micro-memory, and other micro-devices in the field of low energy demand, and has even gradually been expanded to flexible devices and implantable medical devices. Therefore, magnetron sputtering has become the most common method for making LCO thin-film electrodes.

lithium cobalt oxide
thin film
physical vapor deposition

1. Introduction

1.1. Thin-Film Lithium Battery

In recent years, with the rapid development of micro-electromechanical system (MEMS) and smart wearable devices, applicable power sources with high energy density and long cycling life are urgently required [1][2]. E.g., integrated circuits, smart security cards, and other intelligent systems with the micron/nano-scale structures are in need of thin-thickness, light-weight, long-life, high-safety, and high-energy-density power supply. Therefore, the thin-film and miniaturized lithium-ion battery (LIB) has become an important research direction [3][4][5]. Among present cathode materials, lithium cobalt oxide (LCO) has a high working potential and high volumetric capacity [6][7][8][9], and its volumetric change is small enough to avoid film crack upon cycling [10][11][12]. Moreover, the LCO cathode has good conductivity, which can meet the electron transport property without conductive additives. Therefore, the research of the LCO film electrode is important for developing the high-performance thin-film lithium battery (TFB). An effective way to increase the energy density of LCO is to increase its cut-off voltage. The modification methods to elevate the cut-off voltage of powder LCO electrodes are also suitable for thin-film LCO electrodes. However, it faces several problems that hinder its wide application.

1.2. High-Voltage $LiCoO_2$ Cathode

LCO materials have three phases: a high-temperature-phase layered structure (HT-LCO), low-temperature-phase spinel structure (LT-LCO), and rock-salt-phase structure (RS-LTO). The oxygen in the LT-LCO is arranged in an ideal cubic close-packed lattice, and Co^{3+} and Li^+ are distributed on both sides of the oxygen layer, in which the Li^+ layer contains 25% Co^{3+} and the Co^{3+} layer contains 25% Li^+. The Li^+ and Co^{3+} are randomly arranged in the lattice of LCO without a clear boundary. The HT-LCO has excellent electrochemical performance and belongs to the hexagonal R-3m space group with an α-$NaFeO_2$ structure. The oxygen atoms are densely packed with the O3 structure along the (001) crystal plane, occupying the 6c site. The Co^{3+} and Li^+ are alternately arranged occupying the 3b and 3a sites of oxygen octahedral holes.

Although LCO has a theoretical capacity of 274 mAh g^{-1}, its reversible capacity is only approximately 140 mAh g^{-1} (4.2 V vs. Li/Li^+) in practice [13][14][15][16]. Therefore, it is necessary to extract more Li^+ from LCO and maintain the stability of crystal structure [17]. The stability of LCO is affected by several factors: First, when LCO undergoes the deep removal of Li^+, metal-ion migration and cation segregation occur in the crystal lattice, and such structural transformations are irreversible [18][19]. Second, the release of lattice oxygen leads to the collapse of LCO framework, which leads to the distortion of the crystal structure [20][21]. Third, cathode electrolyte interphase (CEI) formation on the surface of LCO affects the Li^+ diffusion and electrode durability. Some reports have noted that the cut-off voltage of LCO should not be above 4.6 V due to the phase transitions at 4.55 V to H1 and H3-3 phases, and the irreversible transformation causes the lithium loss and further capacity attenuation [22][23].

The energy density of TFB using the LCO cathode is restricted by its cut-off voltage, and the modification methods for powder LCO electrodes have important potential for thin-film LCO electrodes [24][25]. Using the surface coating, the formed artificial CEI can inhibit the interfacial side reaction and improve the stability of the LCO film under a higher cut-off voltage for delivering higher reversible capacity [26]. Bulk doping can be achieved by annealing the sputtered LCO film fabricated from a transition metal-doped LCO target under appropriate conditions [27][28]. Both bulk doping and surface coating are beneficial for the electrochemical performance and structural stability of LCO thin films upon cycling.

1.3. Fabrication of $LiCoO_2$ Thin Films

Since the last century, magnetron sputtering deposition technology realizes the merits of high speed, low temperature, and low toxicity. The magnetron sputtering technology perfectly fits the microelectronics, micro-memory, and other micro-devices in the field of low energy demand, and has even gradually been expanded to flexible devices and implantable medical devices [29][30]. Therefore, magnetron sputtering has become the most common method for making LCO thin-film electrodes [31]. Various parameters of magnetron sputtering affect the properties of LCO film electrodes. The growth conditions of sputtered LCO have great influence on the grain orientation, microstructure, and overall stoichiometric ratio of the LCO film, and thus affect its electrochemical performance. Since LCO is an insertion-based cathode material, the powder LCO electrode is usually infiltrated by electrolyte, so that Li can be released from various directions, while the orientation of thin-film LCO material greatly affects the activation energy required for Li migration in all-solid-state batteries [32]. The orientation of LCO films is usually controlled by selecting appropriate substrate materials [33]. When LCO grains nucleate and grow on substrates with specific orientations, they will form the same orientation as the substrate [34][35][36]. Therefore, the optimal orientation conducive to Li-ion transport can be obtained by regulating the substrate [37]. The LCO films with preferred crystal orientation can be obtained by in situ heating or post-annealing at a proper temperature. By controlling the sputtering power, the LCO films with better crystallinity and favorable orientations can also be obtained [38][39][40][41]. At the same time, with the bias voltage, LCO films without post-annealing can be obtained to ensure high crystallization and phase purity [42].

Additionally, LCO thin films can also be fabricated by other techniques, including wet-chemistry [43], atomic layer deposition (ALD) [44], and pulsed laser deposition (PLD) [45]. However, each method has its pros and cons in different application contexts. Wet-chemistry is a good candidate for future mass production, while physical vapor deposition (PVD) is suitable for fabricating thin-film devices. Compared to magnetron sputtering, ALD is good at depositing conformal and ultra-thin LCO films, and PLD can be used to prepare thick LCO films with versatile composition.

2. Sputtering of $LiCoO_2$ Thin Films

According to the characteristics of target materials and the effect of target deposition, magnetron sputtering technology can be divided into radio frequency (RF) sputtering and direct current (DC) sputtering. During operation, secondary electrons are bound near the target surface by the action of a mutually perpendicular electromagnetic field, which increases the ionization rate of gas to a greater extent, thus increasing the density of incident ions. The secondary electrons fall onto the substrate after energy depletion to achieve the low heat and low damage to the substrate. The ionized inert gas molecules bombard the target material under the action of an electromagnetic field to make the materials sputtered onto the substrate, achieving the uniform deposition of films.

2.1. Annealing Process

In contrast to bulk LCO, most LCO films deposited by PVD technology has the problem of insufficient diffusion kinetics for nucleation and growth, which directly leads to the structural instability of LCO films. The annealing treatment of amorphous LCO films in air or oxygen can improve the crystallinity of LCO and reduce the residual stress inside the film. However, along with the increase in crystallinity, the materials are easy to peel from the substrate, and thus the control of annealing parameters is important.

Turrell et al. [46] deposited LCO films on tungsten–nickel alloy foils and annealed them at different temperatures. In the XRD pattern of the film annealed at 700 °C, a peak with orientation of (003) that is not favorable to lithium embedding can be found, while the peak with an orientation of (110) obtained at 500–600 °C disappears at 700 °C. The high temperature brings inevitable changes to the morphology of the substrate, and 600 °C may be the best annealing temperature in terms of structural properties in their work. The maximum capacity of 132 mAh g^{-1} was obtained at 0.1C with a higher cut-off voltage of 4.3 V. However, after 50 cycles, the capacity rapidly decays to 92 mAh g^{-1}, but the cycle stability can be improved at the cost of part reversible capacity. Ma et al. [47] deposited the LCO film directly onto a stainless steel (SS) substrate by RF magnetron sputtering, and compared the influence of annealing parameters on the surface morphology of the LCO film. The annealed LCO films all show network cracks with an average width of ~100 nm. The LCO films annealed in air display more obvious crack with a width of ~400 nm. This phenomenon may be caused by the mismatch of thermal expansion coefficients between the SS substrate and the LCO thin film, leading to the residual stress and peeling from the substrate. Further optimization of the heating rate, holding time, and cooling rate of annealing may achieve the compromise between crystallinity and internal stress. The impurity diffusion from the SS substrate to LCO is also one of the reasons for the damage of the LCO structure. The sample annealed at 500 °C shows an initial discharge capacity of 32.5 μAh cm^{-2} μm^{-1}, but rapidly decreases to 13.4 μAh cm^{-2} μm^{-1} after 37 cycles. This may be caused by the micro-short circuit between thin-film electrodes due to the uneven roughness of the SS substrate.

2.2. Substrate Influence

Unlike powder LCO, the thin-film LCO has the advantage of a large contact area with electrolyte, so that Li$^+$ can be fully extracted without being mainly affected by the grain orientation. However, the contact area between the LCO film and solid electrolyte is also limited when the orientation of LCO film is vertical to the substrate.

Wu et al. [48] found that, for the (003) LCO, Li-ions needs to pass through the Co-O plane with an activation energy of up to 6.8 eV, or must pass through grain boundary with an activation energy of 0.7 eV. This ion diffusion pathway is mainly limited by grain boundary contact at the (003) crystal orientation with high interfacial impedance. Another Li-ion diffusion pathway of (110) crystal orientation is parallel to the electric field, and the Li$^+$ diffusion energy barrier along the CoO$_2$ sheet is as low as 0.3 eV, providing fast Li$^+$ migration channels with the lowest transfer resistance. The TFB using (110) LCO shows an excellent rate performance of ~47 μAh cm^{-2} μm^{-1} (~101 mAh g^{-1}) at a current density of 500 μA cm^{-2} (10C-rates) and remains at 45 μAh cm^{-2} μm^{-1} after 100 cycles at 2C rates. Shiraki et al. [49] prepared the (101¯4) epitaxial LCO films with the CoO$_2$ layer tilted at 52° relative to the surface on the Pt (110) surface. In the laminar LCO, Li-ions mainly diffuse in two dimensions along the CoO$_2$ layer. This means that the (101¯4) and (112¯0) LCO films are suitable for Li-ion conduction. Using the platinum-coated silicon wafers, stainless steels, and titanium plates as substrates, Bohne et al. [50] observed that the different textures of LCO films are highly dependent on the substrate properties rather than the thickness of LCO films. The strong (003) preferred crystal orientation is obtained over a large range of film thickness. However, sputtering LCO films on Si wafers result in the (101) dominant crystal orientation. Yoon et al. [51] improved the electrochemical performance of TFB by adding a Li$_2$O buffer layer to reduce the lattice matching between the LCO cathode and Al (111) current collector. This strategy inhibits the formation of an Li-deficient phase Co$_3$O$_4$ and retards the growth of the unmatched (003) LCO. Although the initial discharge capacity of (003) and (110) LCO cathodes are similar (42.4 and 40.6 μAh cm^{-2}μm^{-1}), the (003) LCO with lower ionic conductivity exhibits obviously decreased capacity as the current density increases.

2.3. Sputtering Power

The post-annealing treatment at high temperature is often accompanied by the reaction between the cathode material and current collecting substrate, which will cause irreversible phase transition upon cycling [52]. Therefore, the crystalline of the LCO film without high-temperature treatment becomes a key problem, which could be mitigated by changing the sputtering power.

Jeon et al. [53] used RF magnetron sputtering to deposit the LCO film on the SS substrate by varying the sputtering power from 50 to 150 W. After deposition, the films show the preferred orientations of (101) and (104). With the increase of sputtering power, the crystallinity of (101) and (104) LCO becomes stronger, which is favorable for improving the lithium transportation. Pan et al. [54] compared the surface morphology of LCO films deposited by different RF sputtering power. The as-deposited films at different power are smooth and crack-free, but the particles become bigger with the increase in sputtering power. After 25 cycles, the capacity retention of LCO films fabricated at 150 and 200 W exceeds 88%, while that of other films prepared at lower power (80 and 100 W) is only ~65%, indicating that increasing the sputtering power can enhance the cycle stability of LCO films. The bias sputtering of LCO is also an effective way to fabricate crystalline LCO films. The enhanced electrochemical properties by the substrate bias are associated with the microscale structural changes due to the ion bombardment, delivering energy to the deposited films. Tintignac et al. [55] obtained crack-free LCO films by a combination of biased sputtering and post-annealing. It is also proven that the de-lithiation process is highly reversible in the LCO films fabricated by biased sputtering.

2.4. Sputtering Atmosphere

Ar is an inert atmosphere to provide the ions for sputtering the target, while oxygen acts as a reactive gas for oxidizing the metal target, thus promoting the generation of the LCO film. Trask et al. analyzed the effect of different O_2 contents on the LCO deposition. The addition of O_2 into the Ar induces a fine grain structure of LCO, resulting into similar morphological changes after high-temperature annealing.

Jason et al. [56] studied the microstructure evolution of LCO films by varying the sputtering atmosphere and noted the reduced grain size and absence of the large cubic grains in the 4% O_2 condition. Without sufficient partial pressure of O_2, when the film is larger than 5 μm, the LCO begins to have large and cubic Li-Co_3O_4 grains with a tightly packed (333) plane parallel to the film. When oxygen is present in the sputtering gas, the LCO film becomes rich in oxygen, thus inhibiting the growth of closed Co_3O_4 grains, and finer grains appear. With the increasing content of O_2, the cycling performance is also gradually increased. The LCO prepared under the conditions of 2%, 3%, and 4% O_2 maintains ~85% of the initial capacity at 0.1C for 100 cycles. The LCO film made under 4% O_2 conditions shows an enhanced cycle performance of 95% capacity retention after 100 cycles. For the pure Ar atmosphere, the L shows single close-packed texture grains. As the O_2 content increases, the as-deposited films become fine grains as a result of the mitigation of volume strain due to the mismatch of LCO and substrate. Tintignac et al. [57] revealed a stratified HT-LCO and cubic LT-LCO in the LCO films and measured the relative content of the two compounds, demonstrating the increase of the layered HT-LCO phase by increasing the gas pressure.

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