Structural and Electrochemical Characterization of Radio Frequency Magnetron-Sputtered \( \text{LiCoO}_2 \) Thin Films

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\( \text{LiCoO}_2 \) thin films are grown by radio frequency magnetron sputter-deposition on platinum-coated \( \text{Al}_2\text{O}_3 \) (c-sapphire) substrates. Deposition parameters such as substrate temperature and the partial pressure ratio between \( \text{O}_2 \) and \( \text{Ar} \) are varied to find suitable parameters for a cathode offering reversible intercalation and deintercalation of lithium ions. Structural characterization is carried out using X-ray diffraction and Raman spectroscopy, revealing that the orientation as well as the crystallinity of the deposited \( \text{LiCoO}_2 \) films can be influenced by varying the \( \text{O}_2/\text{Ar} \) ratio and simultaneously applying a sufficient substrate temperature during deposition. Furthermore, both the atomic composition and the atomic distribution are determined using X-ray photoelectron spectroscopy and secondary ion mass spectrometry. Results show that the films possess surface contaminations of Li and oxygen-rich compounds but have a homogeneous atomic distribution throughout the depth profile. For electrochemical characterization, \( \text{LiCoO}_2 \) thin films where charged and discharged against lithium metal while using a liquid electrolyte consisting of \( \text{LiPF}_6 \) dissolved in a mixture of ethylen carbonate/dimethyl carbonate with a molar fraction of 1:1. Reversible discharge capacities in the order of up to 70 mAh g\(^{-1}\) or 32.40 \( \mu \text{Ah cm}^{-2} \mu \text{m}^{-1} \) can be achieved for optimized \( \text{LiCoO}_2 \) thin films.

1. Introduction

As known, the rechargeable lithium-ion battery (LIB) is the most representative candidate when it comes to powering portable, electrical devices such as cell phones or laptops. \( \text{LiCoO}_2 \) proposed by Goodenough et al., was the first commercially used cathode material in such a LIB. Even today, \( \text{LiCoO}_2 \) is one of the most important cathode materials due to its high energy density (125 Wh kg\(^{-1}\), 440 Wh l\(^{-1}\)) and an achievable battery voltage of over 4 V.\(^{[1]} \) However, due to the ever increasing energy needs of humanity, it is important to further increase the energy density and to continue to ensure or even improve the safety of the batteries. These ideas should be satisfied by the so-called all-solid-state-battery (solid-state battery), a battery made entirely of solid-state materials. Easy miniaturization, no leakage of electrolyte or ignition, and prolonged lifetime are the major advantages that result from the use of a solid electrolyte. In recent years, there has been a severe interest in the application of thin-film cathodes in microelectromechanical systems (MEMS), smart cards, or implantable medical devices. For example, a miniaturized solid-state battery should serve as a so-called “on-chip power supply element” and enable a direct emergency power supply.\(^{[2]} \) \( \text{LiCoO}_2 \) is an extremely promising cathode material due to its excellent electrochemical properties. To date, many studies have focused on \( \text{LiCoO}_2 \) thin-film cathodes fabricated using common techniques such as radio frequency (RF) magnetron sputtering,\(^{[3–5]} \) pulsed laser deposition,\(^{[6,7]} \) spray methods,\(^{[8]} \) sol–gel coating,\(^{[9]} \) and chemical vapor deposition.\(^{[10,11]} \) \( \text{LiCoO}_2 \) crystallizes in the rhombohedral high temperature (HT) or cubic low temperature (LT) phase, depending on the manufacturing conditions. In battery applications, the HT phase is preferred because of its suitable electrochemical properties such as increased capacity and better cycle stability compared with the LT phase. Furthermore, there is a desire to obtain the HT-\( \text{LiCoO}_2 \) film in a suitable orientation in growth direction, as the efficiency of the Li\(^+\) diffusion can be further enhanced. An overview of the properties of RF-sputtered \( \text{LiCoO}_2 \) is given by Julien et al.\(^{[12]} \) In this article, we will investigate the deposition of \( \text{LiCoO}_2 \) thin films via RF magnetron sputtering on heated platinum-coated c-sapphire substrates. Compared with the parameters given by Julien et al., we gain additional information about the thin film using a different substrate and deposition temperature. Structural characterization of the films was carried out as a function of the substrate temperature and the oxygen-to-argon ratio during the deposition. Elemental distribution of atomic compounds near the surface of optimized films was analyzed. Also the electrochemical performance of a \( \text{LiCoO}_2 \) film was evaluated.
2. Results and Discussion

2.1. Structure and Morphology

Figure 1a shows the growth rate of LiCoO₂ depending on the used O₂/Ar ratio at a present substrate temperature of 454 °C during deposition. Film thicknesses were determined using scanning electron microscopy (SEM) images of the breaking edges of the samples, cf. exemplary inset in Figure 1a. An increase in the growth rate (9–20 nm min⁻¹) can be observed when the O₂/Ar ratio is changed from 1:1 to 0:1.

As the working pressure was set constant at ≈0.1 Pa, the mean free path of particles was not influenced. The increasing growth rate with decreasing O₂/Ar ratio can thus simply be attributed to an increased amount of Ar ions, which are mainly responsible for the removal of atoms or clusters out of the LiCoO₂ target. Figure 1b shows a rather small increase in the growth rate (8–9 nm min⁻¹) when the substrate temperature is enhanced from 407 up to 471 °C. Consequently, the increased kinetic energy of the film-forming particles led to increased film thicknesses. According to these results, desorption processes can most likely be excluded. Resulting thicknesses of the thin films for a sputter duration of 1 h at 454 °C are shown in Table 1.

Table 1. Film thicknesses of LiCoO₂ deposited at 454 °C for 1 h, depending on the O₂/Ar ratio used.

| p(O₂): p(Ar) | d (LiCoO₂) [nm] |
|-------------|----------------|
| 1:1         | 524            |
| 1:2         | 561            |
| 1:3         | 590            |
| 1:4         | 653            |
| Ar          | 1195           |

Figure 2a shows the Raman spectra of the LiCoO₂ thin films which were grown for 1 h under the different O₂/Ar partial pressure ratios while applying a constant substrate temperature of 454 °C. Factor-group analysis of the lattice vibrations in

Figure 2. Raman spectra of LiCoO₂ films, a) deposited under different O₂/Ar ratios at 454 °C and b) deposited at different substrate temperatures at a constant O₂/Ar ratio of 1:1.
rhombohedral HT-LiCoO$_2$ with the space group $R3m$ reveals two Raman active vibrational modes, $A_{1g}$ and $E_g$. The $A_{1g}$ and $E_g$ modes are caused by the vibration of oxygen-atom along the $c$-axis and along the $a$–$b$ plane in the LiCoO$_2$ lattice, respectively.$^{[13]}$ As shown in Figure 2a, the $A_{1g}$ and $E_g$ modes of HT-LiCoO$_2$ are observed at 595 and 488 cm$^{-1}$, respectively. With increasing proportion of oxygen in the sputter chamber during deposition, an increase in the modes intensity can be observed. Most likely an oxidation of the target surface occurs, suppressing the formation of Co$_2$O$_4$ and thus favoring the formation of LiCoO$_2$.$^{[14]}$ Also it has to be considered that increasing the oxygen partial pressure can lead to an increase in the Li/Co ratio in LiCoO$_2$, as it was already observed by Liao et al.$^{[15]}$

In addition, a change of the absorption coefficient $\alpha$ takes place when the Li/Co ratio is changed.$^{[13]}$ At a photon energy of 2.41 eV (514 nm), which was used during the Raman experiments, for $x = 0.87$ and $x = 0.53$ in Li$_x$CoO$_2$, the resulting values for $\alpha$ are $0.16 \times 10^6$ and $0.34 \times 10^6$ cm$^{-1}$, respectively.$^{[16]}$ The penetration depth of the laser can then be calculated to 62.5 nm for $x = 0.87$ and 29.4 nm for $x = 0.53$. The minimum film thickness for the examined LiCoO$_2$ films in Figure 2a is 524 nm. Consequently, the absorption coefficient $\alpha$ of LiCoO$_2$ films which were sputtered under high oxygen partial pressure might be increased thus leading to a detection of additional vibrational modes of oxygen atoms in deeper regions of these samples. Figure 2b shows the Raman spectra of LiCoO$_2$ thin films sputtered at different substrate temperatures as well as the spectra of a LiCoO$_2$ thin film postannealed at 700°C. With increasing temperature, an increase in the $A_{1g}$ and $E_g$ modes can be observed, indicating a gain in crystallinity of the thin films. Through the postannealing, the formation of the HT-phase could be further improved. Raman modes of LT-LiCoO$_2$ and Co$_2$O$_4$ as determined experimentally by Mendoza et al. are not observed for the deposited films.$^{[17]}$ In addition to Mendoza et al., a Raman study of LCO thin films is shown by Pan and Yang where a clear difference between the signal structure presented in there and the signal structure obtained from our samples is visible.$^{[18]}$ However, there is no LT-LCO-related Raman mode visible for the very low pressure during deposition like it is known from Tintignac et al.$^{[19]}$ According to the Raman results, LiCoO$_2$ thin films mainly consist of HT-LiCoO$_2$, if a homogeneous growth through the entire depth profile is assumed. While the formation of LT-LiCoO$_2$ during sputtering at a substrate temperature of 454°C seems to be highly unlikely,$^{[20,21]}$ the formation of Co$_2$O$_4$ might not be entirely excluded. A formation of Co$_2$O$_4$ at the interface between the substrate and the LiCoO$_2$ thin films could already be observed$^{[22]}$ and is, because of the low penetration depth of the Raman laser, not detectable with our experimental Raman setup.

Figure 3a shows the X-ray patterns of the LiCoO$_2$ thin films deposited for 1 h under different O$_2$/Ar partial pressure ratios while applying a constant substrate temperature of 454°C. Polycrystalline HT-LiCoO$_2$ and Co$_2$O$_4$ exhibit X-ray reflexes under nearly the same Bragg angles which makes it very difficult to distinguish between these two materials based on X-ray diffraction (XRD) results.$^{[19]}$ According to the Raman results shown, the existence of Co$_2$O$_4$ in deeper regions of the sample can not be excluded. Thus, HT-LiCoO$_2$ as well as Co$_2$O$_4$ were considered for the XRD peak classification. The LiCoO$_2$ thin films show reflexes at angles of 19°, 38.5°, and 59.3°, respectively, which might either be attributed to (003)-oriented HT-LiCoO$_2$ in first, second, and third order or to (111)-, (222)-, and (511)-oriented Co$_2$O$_4$, cf. Figure 3a. Assuming these reflexes originate from Co$_2$O$_4$ might be meaningful especially for the thin films produced with less oxygen or even pure Ar, as a reducing environment can lead to the formation of Co$_2$O$_4$.$^{[14]}$ Nevertheless with increasing the O$_2$/Ar ratio, an increase in the (104) orientation, favorable for LIBs, can be observed. Figure 3b,c shows the influence of the substrate temperature on the (003) and (104) peak intensities when sputtering occurred under an O$_2$/Ar ratio of 1/1. Excluding the film deposited at 407°C, a decrease in (003) and an increase in (104) orientations with rising substrate temperature is observed. Optimized LiCoO$_2$ thin films were repetitively sputtered at a substrate temperature of 454°C and an O$_2$/Ar ratio of 1/1, showing high-intensity Raman modes as well as a mixture of (003)- and (104)-oriented LiCoO$_2$ grains, i.e., a reproduction of the structural properties of the thin films could be achieved. The surface of an optimized film is shown in Figure 4 via SEM (Figure 4a) and via atomic force microscopy (AFM) (Figure 4b), revealing a dense layer of LiCoO$_2$ with atomic
agglomerations with diameters of up to 400 nm on the surface. In a cross section of the thin film, a Volmer–Weber island growth mechanism of LiCoO$_2$ on platinum layer is visible, cf. Figure 4c. This growth mechanism was observed previously for LiCoO$_2$ sputtered onto a platinum layer.\[23\] The surface agglomerations might be a consequence of the island growth mechanism and could be reduced with a reduction of the sputtering power and thus the growth rate. Yet, an average surface roughness of 8.2 nm could be estimated for the optimized LiCoO$_2$ film.

2.2. Phase Determination via X-Ray Photoelectron Spectroscopy

The X-ray photoelectron (XP) spectra of an optimized thin film are shown in Figure 5. In addition to the XP signal of the surface, three additional spectra were recorded, each after 120 s of Ar$^+$ etching. Figure 5a shows the Co 2p core levels with its components $2p_{3/2}$ and $2p_{1/2}$ at binding energies of 780 and 795.5 eV which arise as a result of the spin–orbit coupling. The accompanying satellite structures at 789 and 804 eV can be attributed to Co$^{3+}$, as it is expected for LiCoO$_2$. The intensity of the Co 2p photoelectron signal is increased continuously when Ar$^+$ etching was accomplished, indicating the presence of phases without Co on the surface of the film. The O 1s spectrum of the film exhibits two peaks labeled $O^{2-}$ surface and $O^{2-}$ bulk at binding energies of 529 and 531.6 eV. $O^{2-}$ bulk can be attributed to lattice oxygen in the LiCoO$_2$ network, whereas $O^{2-}$ surface arises as a consequence of oxygen containing surface species such as LiCoO$_2$, LiOH, and Li$_2$O.\[24\] The Li 1s signal of the LiCoO$_2$ surface in Figure 5c occurs at a binding energy of 55 eV, whereas the Li 1s photoelectron of LiCoO$_2$ is expected to arise at binding energies of $\approx$54 eV.\[25,26\] As already described for the O 1s signal, species Li$_2$CO$_3$, LiOH, and Li$_2$O can be obtained within the Li 1s signal in the surface region of the thin film, shifting the signal to higher binding energies. For the C 1s spectrum of the analyzed film, three different types of carbon-containing
species can be observed. A strong peak arises due to the contamination of the surface by aliphatic compounds at 284.8 eV. Furthermore, a contamination of esters as well as the already specified compound Li$_2$CO$_3$ are observed at binding energies of 288.9 and 289.6 eV, respectively. In summary, the LiCoO$_2$ thin films posses surface contaminations of Li$_2$CO$_3$, LiOH, and Li$_2$O which are still detectable after 360 s of Ar$^+$ etching, implying that just a few nanometers of the thin film were removed by the etching process. These circumstances hinder a meaningful evaluation of the atomic concentration of Li, Co, and O in the LiCoO$_2$ thin film. Nevertheless, mass spectrum of the thin film was recorded via secondary-ion mass spectrometry (SIMS) to obtain the atomic relation between Co and Li, revealing a homogeneous distribution of Li and Co through the entire depth profile of the sample (not shown here).

2.3. Electrochemical Characterization

The measured cyclovoltammogram of the electrochemical half cell containing the optimized LiCoO$_2$ thin film as a positive electrode is shown in Figure 6. The characteristic oxidation peak during charging can be observed at a voltage of 3.92 V versus Li/Li$^+$. The reduction peak during the discharge occurs at 3.88 V, which yields a peak separation of about 40 mV characteristic for HT-LiCoO$_2$.[27]

The charge and discharge capacities of the electrochemical half cell containing the optimized LiCoO$_2$ thin film are shown in Figure 7. For calculating the mass of the 524 nm-thick LiCoO$_2$ film, the gravimetric density was estimated to be 4.5 g cm$^{-3}$ (90% of the literature value). Consequently, the used specific current of 11.6 μA cm$^{-2}$ corresponded to a charge rate of 0.35. Figure 7a shows the charge curves of the cell for the first 20 cycles. The charge plateau is found to be at 3.9 V. The charge capacities reached for the different cycles can be read off at the intersections between curves and the upper axis of abscissae in units of mAh/g. While upon the first charge, a capacity of 72 mAh g$^{-1}$ or 32.4 μAh cm$^{-2}$ μm$^{-1}$ can be observed, the capacity continuously faded by 0.7% per cycle down to a value of 62 mAh g$^{-1}$ or 27.9 μAh cm$^{-2}$ μm$^{-1}$ for the 20th cycle. Discharging the cell for the first time delivered a discharge capacity of 70 mAh g$^{-1}$ or 31.5 μAh cm$^{-2}$ μm$^{-1}$. Cycling of the cell delivered a capacity fading of 0.7% per cycle, similar to the behavior of the charge capacity. The corresponding Coulomb efficiency is ~93% for every charge/discharge cycle. The obtained discharge capacities of the first cycle are higher than for a sputter power optimized LCO thin film like it is shown in Jan et al.[28] Although the discharge capacities in Zhu et al. are quite higher than for the thin films presented in this work, the Coulombic efficiency of 93% is higher in this case.[29] However, the capacity fading in the publication of Zhu et al. is even bigger (~3.7%) than for the films reported in here (~0.7%). Most likely, this capacity fading is related to a degradation of the electrolyte used and might be further reduced by lowering this degradation.

![Figure 6. Cyclovoltammogram of the optimized LiCoO$_2$ thin film. The cyclovoltammogram was recorded using a sweep rate of 1 mV s$^{-1}$.](image)

![Figure 7. a) Charging and b) discharging curves of an electrochemical half cell including the optimized LiCoO$_2$ thin film. The cell with the sequence LiCoO$_2$|separator + LiPF$_6$ in EC/DMC|Li was cycled over the voltage range of 3.5–4.2 V at a specific current of 11.6 μA cm$^{-2}$ or rather 0.35 C.](image)
addition, to the discharge curves of the optimized LiCoO$_2$ thin film. Figure 7b shows an inset showing the discharge curves of LiCoO$_2$ sputtered under different substrate temperatures as well as a postannealed LiCoO$_2$ film. One can observe that the substrate temperature leads to an increase in the discharge capacity. This can most likely be understood in terms of the higher crystallinity of these films, which allows a more efficient intercalation and deintercalation of Li$^+$ within the LiCoO$_2$ network. The highest discharge capacity of 93 mAh g$^{-1}$ or 41.85 $\mu$Ah cm$^{-2}$ $\mu$m$^{-1}$ can be found for the electrochemical cell including the postannealed LiCoO$_2$ thin film. By post annealing a further increase in discharge capacity due to a almost higher crystallinity of the deposited films can be observed. So one has to decide if a higher deposition temperature or a postannealing step is suitable for production of high discharge capacity LCO material. If one compares the specific capacity of this work to the specific capacity of Tintignac et al.,$^{[19]}$ the values obtained in our experiments are quite lower. This can be related to the fact that the deposition of our thin films was done at a working pressure of 0.1 Pa. An increase in specific capacity is related to an increase in pressure during deposition so the specific capacity of Tintignac et al. has to be higher because of the higher pressure during deposition ($\approx$ 3 Pa). Tintignac et al. measured a specific capacity of 52 $\mu$Ah cm$^{-2}$ $\mu$m$^{-1}$ for 3 Pa and 22 $\mu$Ah cm$^{-2}$ $\mu$m$^{-1}$ for a deposition pressure of 0.55 Pa. However, the obtained value of 32.4 $\mu$Ah cm$^{-2}$ $\mu$m$^{-1}$ at 0.1 Pa and 454 °C for the results presented in here is higher than the results shown in the study by Tintignac et al.$^{[19]}$ at an increased deposition pressure.

Regarding the review on RF-sputtered LCO thin films by Julien et al., a new substrate/thin-film combination at different substrate temperatures and Ar/O$_2$ ratios was tested, obtaining a moderate discharge capacity of about 32.4 $\mu$Ah cm$^{-2}$ $\mu$m$^{-1}$ for the films deposited at substrate temperature of 454 °C and 41.85 $\mu$Ah cm$^{-2}$ $\mu$m$^{-1}$ for the postannealed films.$^{[12]}$ The experimental conditions and results are finally shown in Table 2.

### Table 2. Deposition parameter and properties of the LCO thin films obtained in this work.

| $p$(O$_2$):$p$(Ar) | $p$ [W cm$^{-1}$] | $d_{CO}$ [nm] | $T$ [°C] | Substrate | Discharge capacity | Structure |
|------------------|------------------|---------------|----------|-----------|------------------|-----------|
| 1:1              | 2.2              | 524           | 454      | Al$_2$O$_3$/Pt | 32.40 $\mu$Ah cm$^{-2}$ $\mu$m$^{-1}$ | (104)-orientation |
| 1:1              | 2.2              | 524           | 700 (post annealed) | Al$_2$O$_3$/Pt | 41.85 $\mu$Ah cm$^{-2}$ $\mu$m$^{-1}$ | (104)-orientation |

As a result of this growth mechanism, particle agglomerations with diameters of up to 400 nm on the sample surfaces were observed. The average surface roughness was calculated to be 8.9 nm via AFM. XP spectra revealed impurity phases in the surface region such as Li$_2$CO$_3$, Li$_2$O, and LiOH. After surface etching with Ar$^+$ ions, however, the signal Li 1s could be clearly assigned to LiCoO$_2$. Electrochemical half cells with the layer sequence LiCoO$_2$ || LiPF$_6$ in ethylene carboxylatedimethyl carbonate (EC:DMC 1:1 || Li) were produced and successfully charged and discharged in a voltage range of 3.5–4.2 V versus Li/Li$^+$. While cyclic voltammetry (CV) measurements approved the presence of HT-LiCoO$_2$, cycling the cell at a specific current of 11.6 $\mu$A cm$^{-2}$ or rather 0.35 C led to discharge capacities of 70 mAh g$^{-1}$ or 31.5 $\mu$Ah cm$^{-2}$ $\mu$m$^{-1}$. A fade of the capacity of 0.7% per cycle was observed leading to a remaining capacity of 60 mAh g$^{-1}$ or 27 $\mu$Ah cm$^{-2}$ $\mu$m$^{-1}$ after 20 cycles. Coulombic efficiency was observed to be constant at $\approx$93%.

### 4. Experimental Section

LiCoO$_2$ thin films were produced via RF magnetron sputtering. Stoichiometric LiCoO$_2$ with a purity of 99.7% from the company Kurt J. Lesker was used as sputtering target. c-Sapphire (Al$_2$O$_3$) was coated with a dense layer of 100 nm platinum and was used as a substrate. The platinum layer served as a current collector and was realized by an RF sputtering process of a metallic platinum target using 200 sccm Ar and a power density of 0.55 W cm$^{-2}$. For the deposition of the LiCoO$_2$ thin films, the substrate deposition temperatures (407, 432, 454, and 471 °C), as well as the partial pressure ratio between the O$_2$ reactive gas and the Ar sputter gas (0:1, 1:1, 1:2, 1:3, and 1:4) were varied. A sputtering power density of 2.2 W cm$^{-2}$ was applied to the LiCoO$_2$ target. In a prior work, the sputtering power and working pressure were determined as sufficient for these values. Also the optimal temperature range for our setup was identified at about 400–460 °C. If one considers the temperature range of Zhu et al.$^{[29]}$ the temperature range in this work seems reasonable. The gas working pressure during deposition was set constant to 10$^{-3}$ mbar. The substrate temperature was measured in advance, contacting the platinum surface of the substrates with a thermocouple while heating to the desired values. Raman spectroscopy was used to specify the thin films possible structural components such as LT-LiCoO$_2$, HT-LiCoO$_2$, and Co$_3$O$_4$. Light with the wavelength of 514 nm was produced by an Ar-ion laser and focused to a 15.9 $\mu$m-sized spot on the thin-film surfaces, resulting in a power density of $\approx$84 mW $\mu$m$^{-2}$. The crystalline phase and the orientation of the produced thin films were examined by XRD in Bragg–Brentano geometry using a Siemens Bruker D5000. Aluminum K$_\alpha$ radiation (K$_\alpha$ = 1486 eV) was accelerated on the films surfaces, whereas the angle of incidence was changed with a rate of 2°/min. Information concerning the chemical bonds were determined via X-ray photoelectron spectroscopy (XPS) using a PHI VersaProbe. X-rays were produced using an Al-anode (K$_\alpha$ = 1486 eV). Measurements were carried out with a source angle of 45°. All experiments were realized with charge neutralization on the sample surface, and all spectra were referenced to the carbon signal (C 1s) at 284.8 eV. Depth profiles were measured via in situ argon-ion etching with acceleration voltage of 1 kV. Atomic distribution of the films was determined by SIMS. The surface morphology, cross section, and thus the

### 3. Conclusion

The RF magnetron sputter deposition of LiCoO$_2$ thin films was successfully optimized by adjusting the deposition parameters such as substrate temperature and the oxygen-to-argon ratio in the plasma. Using a substrate temperature of 454 °C and adding oxygen as a reactive gas to the deposition, the formation of rhombohedral HT-LiCoO$_2$ as well as (104)-oriented LiCoO$_2$ grains could be established. Best results were obtained for an O$_2$/Ar partial pressure ratio of 1:1 and a substrate temperature of 454 °C, in terms of the most intense A$_{1g}$ and E$_{g}$ modes in Raman spectra and an increased proportion of (104)-oriented grains. Based on SEM images of the cross section of the samples, a Volmer–Weber growth of LiCoO$_2$ on platinum was detected.
Figure 8. Schematic representation for the construction of electrochemical half cells with the sequence LiCoO$_2$|separator + LiPF$_6$ in EC/DMC|Li.

thickness of thin films were examined by SEM using a Zeiss-Merlin setup. Surface roughness was evaluated utilizing AFM. For LiCoO$_2$ cathodes with a defined electrode area, substrates were provided with a temperature stable ceramic mask during the LiCoO$_2$ sputter deposition, resulting in electrode areas of 6 × 5 mm$^2$. Electrochemical half cells were then assembled following the scheme in Figure 8. Fiberglass filters served as a separator and 1 M LiPF$_6$ in EC/DMC (1:1 by vol%) was used as the electrolyte. The charge/discharge testing and CV measurements were carried out using a VMP300 potentiostat. The cells were cycled over the voltage range of 3.5–4.2 V at a specific current of 11.6 μA cm$^{-2}$ or rather 0.35 C. For CV measurements, a sweep rate of 1 mV s$^{-1}$ was used.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

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