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Effect of deposition conditions on the electrochemical characteristics of SnO_2 thin films as anodes for lithium-ion batteries

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Abstract: SnO_2 thin-film anodes were fabricated by RF magnetron sputtering of a metallic Sn target onto Ti–Pt coated stainless-steel substrates. The films were deposited at room temperature (RT) and 300°C in an Ar/O_2 atmosphere and subsequently annealed in air at 620°C. The structural evolution, morphology, and electrochemical performance of the films were systematically investigated. X-ray diffraction (XRD) analysis confirmed the formation of phase-pure tetragonal rutile SnO_2 without detectable secondary phases. SEM observations revealed significant changes in grain size and surface uniformity depending on deposition temperature and thermal treatment. Electrochemical testing in CR2032 coin cells demonstrated a typical multistep lithiation mechanism of SnO_2 involving conversion and alloying reactions. Although annealing improved crystallinity and reduced polarization, the films deposited at RT exhibited superior long-term cycling stability compared to those deposited at elevated temperature. The results highlight the crucial role of deposition temperature and sublayer configuration in controlling structural integrity and electrochemical durability of SnO_2 thin-film anodes for lithium-ion batteries.

Keywords: Lithium-ion batteries, SnO_2, Ti/Pt interlayer, RF magnetron sputtering, annealing, electrochemical performance.

1. Introduction

Lithium-ion batteries (LIBs) have become the cornerstone of modern

electrochemical energy storage systems due to their high energy density, long cycle life, and relatively low self-discharge rate (Manthiram, 2020). Their widespread implementation in portable electronics, electric vehicles, and grid-scale energy storage has accelerated the demand for electrode materials capable of delivering higher capacity, improved rate capability, and long-term cycling stability (Nanda et al., 2015). Although graphite remains the dominant commercial anode material, its theoretical capacity of 372 mAh g^{-1} limits further enhancement of overall battery energy density (Cameán et al., 2010).

To overcome this limitation, significant research efforts have been directed toward alternative anode materials with higher theoretical capacities, including silicon, transition metal oxides, and conversion-type compounds (Poizot et al., 2000; Cabana et al., 2010). Among these, tin-based materials have attracted particular attention due to their favorable electrochemical properties and relatively high theoretical capacity (Kamali et al., 2011). Tin oxide (SnO_2) is a promising anode material for LIBs due to its high theoretical capacity (1494 mAh g^{-1}), which is significantly higher than that of metallic tin, oxidation resistance, and environmental safety. The promising characteristics of SnO_2 in LIBs are explained not so much by its high theoretical capacity as by the peculiarities of the two-stage mechanism of lithium accumulation (Yegamkulov et al., 2025). At the first stage, a transformation reaction occurs:

$$SnO_2 + 4Li^+ + 4e^- \leftrightarrow Sn + 2Li_2O \quad (1)$$

and at the second stage, Sn alloying takes place

$$Sn + xLi^+ + xe^- \leftrightarrow Li_xSn \quad (2)$$

At relatively moderate potentials (1.2 V and 0.5 V, respectively, relative to Li/Li^+). These potentials reduce the likelihood of lithium plating on the anode surface and help to suppress polarization. Thus, the high electrical conductivity of the lithiated phases, the short diffusion length of Li^+, and the ability of SnO_2 to form stable nanostructures determine its effectiveness as an anode material in LIBs (Serikkazyyeva et al., 2023). The combination of conversion and alloying reactions enables high capacity but is accompanied by severe volume expansion (up to 300%) during cycling (Asenbauer et al., 2020). Such large volume fluctuations generate internal stress, leading to particle pulverization, loss of electrical contact, unstable solid electrolyte interphase (SEI) formation, and rapid capacity fading (Yu et al., 2007).

Various strategies have been proposed to address these limitations. Nanostructuring of SnO_2 reduces diffusion lengths for lithium ions and improves strain accommodation during cycling (Kebede, 2020). Additionally, composite formation with conductive matrices such as carbon or metallic scaffolds enhances electronic conductivity and structural stability (Huang et al., 2016). Another promising approach involves the design of thin-film electrodes, which provide better mechanical integrity and strong adhesion to the current collector (Teng et al., 2020).

Thin-film anodes are particularly attractive for fundamental studies and microbattery applications (Omampuliyur, 2015). Compared to bulk powder electrodes, thin films eliminate the need for binders and conductive additives, allowing direct evaluation of intrinsic electrochemical properties. Furthermore, thin films exhibit shorter lithium diffusion pathways and improved interfacial contact, which can enhance rate performance and structural stability (Xia et al., 2023).

Among various thin-film fabrication techniques, RF magnetron sputtering offers precise control over thickness, composition, and microstructure (Nur-E-Alam et al., 2019). Deposition parameters such as substrate temperature, working pressure, and gas composition strongly influence crystallinity, grain size, defect concentration, and surface morphology. These microstructural factors directly affect lithium diffusion kinetics, charge-transfer resistance, and mechanical stability during cycling (Ren et al., 2025).

Deposition temperature is a particularly critical parameter. Films deposited at room temperature often exhibit amorphous or nanocrystalline structures, which can better accommodate volume changes

due to their structural flexibility (Kumar et al., 2013). In contrast, films deposited at elevated temperatures generally display higher crystallinity and larger grain size, which may enhance electronic transport but reduce tolerance to mechanical stress during repeated lithiation/delithiation cycles (Ghantasala et al., 2023). Post-deposition annealing is commonly applied to improve crystallinity and structural ordering; however, excessive grain growth may negatively affect electrochemical durability (Morankar et al., 2024).

In addition to the active material itself, the interface between the thin film and the current collector plays a crucial role in electrode stability. The use of adhesion-promoting and conductive interlayers, such as Ti and Pt, can improve mechanical bonding, reduce interfacial resistance, and suppress delamination during cycling (Shao et al., 2017). Engineering such sublayer configurations is therefore essential for optimizing thin-film electrode performance.

This work is devoted to the systematic study of the electrochemical characteristics of SnO_2 thin-film anodes obtained by radio frequency (RF) magnetron sputtering at room temperature (RT) and at 300 °C. The as-deposited films were subsequently subjected to post-annealing treatments in air, in order to investigate the influence of deposition conditions and heat-treatment environments on their structural evolution and electrochemical performance.

2. Methodology

2.1. Material preparation

SnO_2 anode thin films were deposited by RF magnetron sputtering (Angstrom Engineering Inc., Canada) of a high-purity Sn metallic target (2.00'' diameter × 0.250" thick, 99.994-99.999% pure, Kurt J. Lesker Company) in an Ar/O_2 (high purity, 15:15 sccm) atmosphere. Stainless steel (SS) discs served as substrates, which were precoated with a thin Ti-Pt bilayer to improve adhesion and provide a stable interface. The depositions of anode thin films were carried out at a working pressure of 5 mTorr and a power of 60 W, both at RT and at 300 °C, with substrate rotation to ensure uniformity. The resulting film thickness was about 500 nm. Prior to deposition, the Sn target surface was cleaned by pre-sputtering in pure Ar and subsequently in an Ar/O_2 mixture for 20 minutes under the same plasma conditions, while the substrate was shielded. This step ensured the removal of surface oxides and contaminants from the target.

After sputtering, the films were annealed at 620 °C in air to promote crystallization and modify the surface morphology. Film thickness and mass were monitored using an INFICON Quartz Crystal Monitor (QCM) and an ultra-microbalance (Mettler Toledo XP2U Ultra Microbalance).

2.2 Material characterization

To study the structure and morphology of the samples, their comprehensive physicochemical characterization was carried out using various analytical methods. Morphological analysis was carried out using scanning electron microscopy (SEM, JEOL JSM- IT800, Japan). The crystal structure was studied by X-ray diffraction (XRD) on a SmartLab (Rigaku) setup using Cu Kα radiation ($\lambda_{CuK\alpha1}$ = 1.54056 Å, $\lambda_{CuK\alpha2}$ = 1.54439 Å) in the Bragg-Brentano reflection geometry. The XRD data were obtained over a 2θ range from 5 to 70 °C at a scan rate of 5 °C/min using 40 kV, 30 mA X-ray.

2.3 Electrochemical investigation

CR2032 coin cells were assembled with the obtained thin film SnO_2 anodes and tested. A Celgard 2400 polypropylene membrane was used as a separator and lithium foil as an opposite and reference electrode. The assembly was carried out in an Ar-filled glove box (MBRAUN LABMaster Pro, Germany) in a high-purity argon atmosphere with <0.1 ppm O_2 and <0.1 ppm H_2O to avoid moisture and oxygen contamination. A 1M $LiPF_6$ solution in an EC/DEC/EMC mixture (1:1:1 by volume) was used

as the electrolyte.

Electrochemical testing was performed using cyclic voltammetry (CV, VMP3, BioLogic, France) and galvanostatic charge-discharge cycling with a multichannel battery testing system (Neware Co., Shenzhen, China). The system's long-term cycling performance was evaluated at a 0.1 °C rate within a potential range of 0.01 to 3 V *vs.* Li/Li^+.

3. Results

In this study, SnO_2 thin films were produced by RF magnetron sputtering of a metallic Sn target onto Ti and Pt-coated stainless steel (SS) substrates. The process was carried out in an Ar/O_2 gas mixture (15:15 sccm) at a constant pressure of 5 mTorr. Both amorphous and crystalline SnO_2 were examined, since amorphous films are usually considered more flexible and better able to handle the volume changes that occur during lithiation and delithiation. The deposition was carried out either at RT and at 300 °C to see how deposition temperature affects their structure, morphology, and electrochemical behavior. Post-deposition annealing was carried out in air at 620 °C to enhance structural ordering and improve surface morphology.

3.1. XRD analysis

The X-ray diffraction (XRD) patterns of the SnO_2 thin films (Figure 1) confirm the significant influence of both deposition temperature and post-annealing atmosphere on the structural evolution of the films.

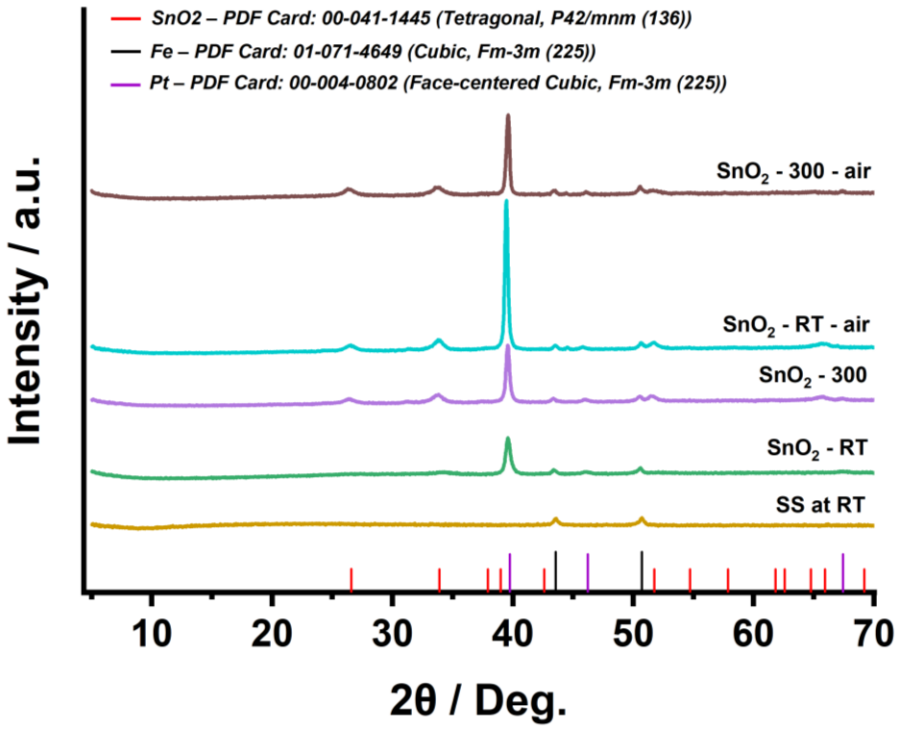

Figure 1. XRD patterns of SnO_2

All coated samples exhibit diffraction peaks corresponding to tetragonal rutile SnO_2, confirming the formation of a crystalline oxide phase. In addition to the oxide reflections, two peaks from the stainless-steel substrate (α-Fe, cubic) are observed at approximately 44.7° and 65.0°, corresponding to

the (110) and (200) planes, respectively. Two peaks attributed to Pt from the Ti-Pt sublayer are detected at about 39.8° and 46.2°, corresponding to the (111) and (200) planes of face-centered cubic Pt. These metallic reflections arise from the underlying layers and do not indicate the formation of secondary phases within the SnO_2 coating (Courtney et al., 1997).

No distinct diffraction peaks corresponding to Ti are detected. This can be explained by the structural configuration of the sublayer: Ti was deposited first, and its thickness is approximately two times smaller than that of the Pt layer. As a result, the thinner Ti layer is effectively shielded by the thicker Pt overlayer and the SnO_2 film, significantly reducing its diffraction intensity.

The dominant SnO_2 reflections appear at 26.6° (110), 33.9° (101), 51.8-54.8° (211), and 61.9–65.9° (301)/(310), confirming the stability of the rutile structure after air annealing. The most intense peak near 39-40° is mainly associated with the SnO_2 (200) plane, partially overlapping with the Pt (111) reflection (Habte et al., 2020).

No additional peaks corresponding to secondary phases such as SnO or metallic β-Sn are detected.

The XRD patterns confirm high phase purity of the films, with variations in peak intensity primarily related to differences in crystallinity and preferred orientation, as well as limited overlapping from the Pt interlayer and Fe substrate.

3.2 Scanning electron microscopy

SEM images (Figure 2) show the morphology of SnO_2 films under different deposition and annealing conditions.

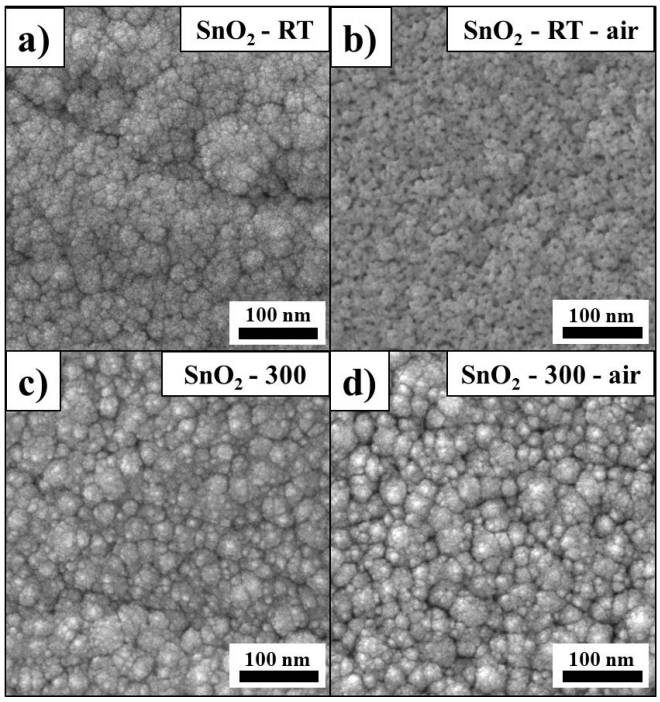

Figure 2. SEM surface images of SnO_2 (a) as-deposited at RT, (b) post-annealed in air, (c) as-deposited at 300 °C (d) post-annealed in air

The morphology and crystallinity of the as-deposited SnO_2 films are strongly influenced by the deposition temperature. The SnO_2–RT film (Figure 2a) exhibits a dense, nanogranular structure composed of very fine, uniformly distributed particles, which is consistent with XRD analysis showing

broad and weak diffraction peaks indicative of low crystallinity and small grain size. In contrast, the SnO_2–300 film (Figure 2c) displays a more defined granular texture and improved structural uniformity, reflected by sharper and more intense XRD peaks, suggesting enhanced crystallinity and significant grain growth at the elevated substrate temperature.

Air annealing further improves structural ordering. SnO_2–RT–air (Figure 2b) becomes smoother and more homogeneous due to pronounced crystallization and grain coarsening, as confirmed by stronger and narrower XRD peaks. SnO_2–300–air (Figure 2d) exhibits more subtle morphological changes; however, additional grain growth and improved uniformity are still observed, consistent with slight peak sharpening in the diffraction patterns. Overall, air annealing promotes crystallization and structural refinement while preserving the rutile SnO_2 phase composition.

4. Discussion

The prepared thin-film electrodes were tested in lithium cells using galvanostatic cycling. The charge/discharge voltage curves of SnO_2-based anodes (Figure 3a and b) represent a comprehensive assessment of the electrochemical characteristics of these electrodes. This figure shows profiles for electrodes treated under various conditions, including after deposition at RT and after subsequent annealing in air, as well as for electrodes deposited at an elevated temperature of 300°C under similar conditions after annealing treatment conditions.

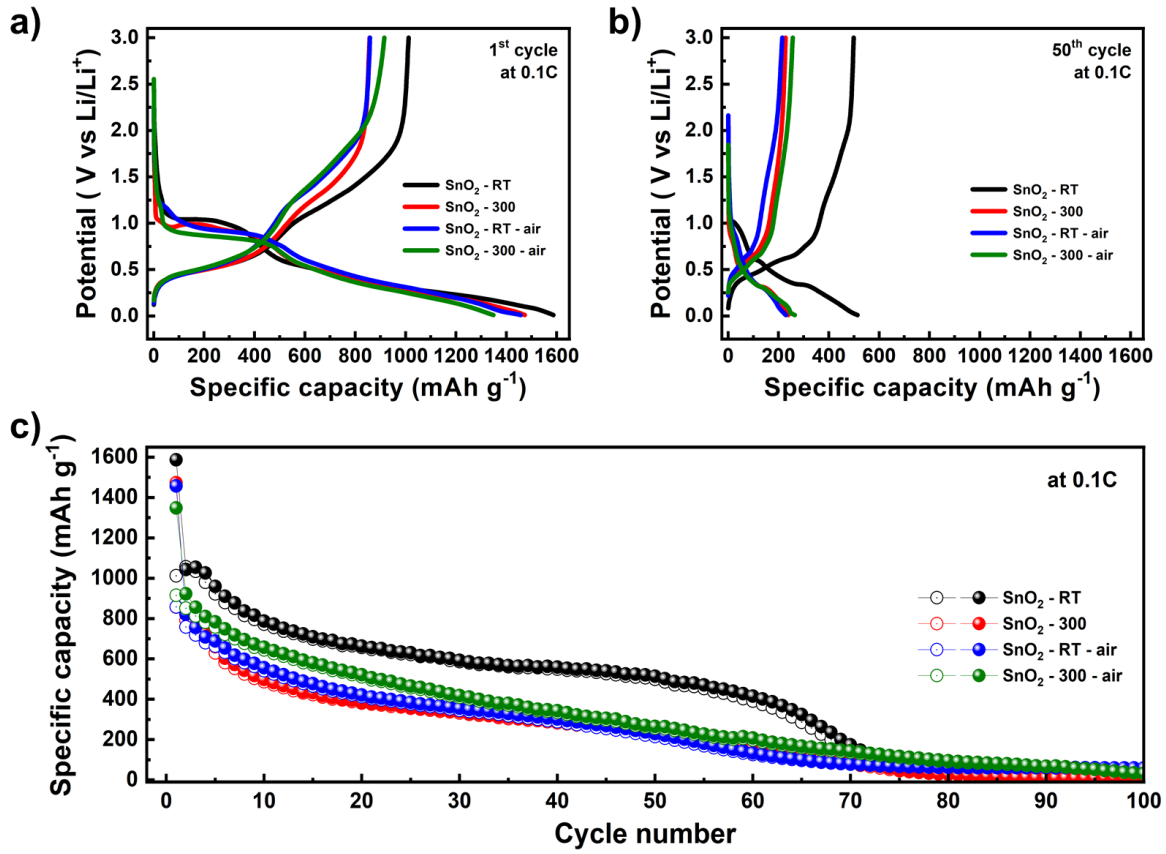

Figure 3. Potential profiles of all samples at 0.1C in (a) 1st and (b) 50th cycles and (c) cycle performance of SnO_2 anodes at 0.1C

The first-cycle discharge/charge curves (Figure 3a) exhibit the typical multistep lithiation mechanism of SnO_2. During the initial discharge, all samples show a broad sloping region above 1.0-1.2 V, which corresponds to the irreversible conversion of SnO_2 to metallic Sn, accompanied by the formation of Li_2O (Dong et al., 2017). This step is associated with large polarization, especially in the SnO_2–RT sample, as indicated by the more pronounced potential drop and broader reaction region. In contrast, the annealed samples, particularly those treated at 300 °C, demonstrate reduced polarization and more well-defined conversion plateaus, reflecting improved charge-transfer kinetics. The second characteristic region in the first discharge cycle appears at 0.5-0.8 V, corresponding to the alloying of Sn with Li to form Li_xSn phases (Serikkazyyeva et al., 2023). The alloying plateau is clearer and occurs at lower overpotential for the annealed samples, again indicating lower polarization. Below 0.2 V, all samples show additional capacity associated with deep lithiation and SEI formation, contributing to the large initial irreversible capacity loss (Zhang et al., 2019).

By the 50th cycle (Figure 3b), the potential curves become more stable and reproducible. The hysteresis between discharge and charge curves decreases for all samples, particularly those thermally treated. The main reversible plateau around 0.4-0.8 V, corresponding to Li_xSn alloying/dealloying, becomes the dominant feature (Jiang et al., 2017). SnO_2–RT maintains the highest polarization even after cycling, displaying larger potential gaps and a less distinct plateau, indicating slower kinetics and structural degradation. Meanwhile, the SnO_2–300 sample preserves clearer plateaus and lower overpotentials, demonstrating improved structural and electrochemical stability during repeated cycling.

The comparison is based on deposited and post-annealed samples of SnO_2 (Figure 3c). The sample SnO_2–RT shows the best cycling stability compared to all other conditions, including annealed samples. In contrast, the sample SnO_2–300 shows a sharp decrease in capacitance and poor capacitance retention, similar to the behavior observed for the Ti-only sublayer sample (Yegamkulov et al., 2025). This emphasizes the negative effect of elevated deposition temperature on the lifetime of the coatings. The sample SnO_2–RT–air shows good initial behavior but exhibits a gradual decrease in capacitance, not surpassing the stability of the untreated sample at RT. In all cases, deposition at 300 °C results in worse capacity retention, indicating thermal instability or enhanced side reactions at elevated temperatures. Thus, the sample deposited at RT stands out as the most stable among all tested conditions for SnO_2 with a Ti–Pt sublayer. Probably, the presence of Pt in the sublayer contributes to the stabilization of the structure even without high-temperature annealing, in contrast to the Ti-only sublayer described in the work (Yegamkulov et al., 2025).

5. Conclusion

SnO_2 thin films deposited by RF magnetron sputtering with a Ti-Pt interlayer exhibit electrochemical properties strongly dependent on deposition and annealing conditions. XRD and SEM analyses confirm that annealing in air promotes crystallization and formation of a porous nanostructure that enhances Li^+ transport and mechanical integrity during cycling. In particular, it is found that amorphous films deposited at room temperature exhibit higher cycling stability due to their structural flexibility and ability to buffer volume expansion during lithiation/delithiation. In contrast, crystalline films after annealing show a higher degree of crystallinity and reduced polarization; however, they demonstrate poorer long-term cycling stability as a result of grain growth and decreased mechanical stability. Films deposited at room temperature demonstrate the most stable performance over first few cycles, compared to other samples. A gradual capacity decay is observed for all samples up to the 100^{th} cycle. These results indicate that the optimal configuration for stable and high-performance SnO_2 anodes includes deposition at room temperature and the use of a Ti-Pt interlayer to improve interface stability and conductivity and demonstrate how the electrochemical performance of SnO_2 thin film anode evolves when a Ti-Pt interlayer is used, in comparison with a single Ti interlayer. This study contributes to the

optimization of SnO_2-based thin film anodes for advanced lithium-ion microbattery technologies.

6. Supplementary Materials: No Supplementary Materials.

7. Author Contributions

Conceptualization - Zh.B., M.Y., A.B.; methodology - Zh.B., M.Y., A.B.; formal analysis - Zh.B., M.Y.; investigation - M.Y.; data curation - M.Y., G.T., A.N., A.M.; writing-original draft - Zh.B.; writing - review & editing - Zh.B., M.Y., A.B.; visualization - Zh.B., M.Y., A.B.; supervision - G.N., A.N., Z.Bk., A.M.; project administration - A.N., A.M.

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11. Conflicts of Interest: No conflicts of interest are declared by the authors.

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SnO_2 жұқа пленкалы литий-ионды аккумуляторлардың аноды ретінде электрохимиялық сипаттамаларына шашырау шарттарының әсері

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Аңдатпа. SnO_2 жұқа пленкалы анодтары Ti–Pt аралық қабатымен қапталған тот баспайтын болат (SS) субстраттарына метал Sn нысанасын радиожиілікті (РЖ) магнетронды шашырату әдісі арқылы алынды. Пленкалар Ar/O_2 атмосферасында бөлме температурасында (RT) және 300°C-та тұндырылып, кейіннен 620°C-та ауада күйдірілді. Пленкалардың құрылымы, морфологиясы және электрохимиялық қасиеттері жүйелі түрде зерттелді. Рентгендік дифракциялық талдау (XRD) нәтижелері бөгде фазаларсыз тетрагональды рутил құрылымды таза SnO_2 фазасының түзілгенін растады. СЭМ (SEM) зерттеулері тұндыру температурасы мен термиялық өңдеуге байланысты түйіршік өлшемі мен беттік біртектіліктің айтарлықтай өзгеретінін көрсетті. CR2032 типті тиын элементтерінде жүргізілген электрохимиялық сынақтар SnO_2 үшін конверсиялық және легірлеу

реакцияларын қамтитын көпсатылы литийлену механизмін анықтады. Күйдіру кристалдылықты арттырып, поляризацияны төмендеткенімен, бөлме температурасында тұндырылған пленкалар жоғары температурада алынған үлгілермен салыстырғанда ұзақ мерзімді циклдік тұрақтылықтың жоғары көрсеткішін көрсетті. Алынған нәтижелер SnO_2 жұқа қабатты анодтарының құрылымдық тұтастығы мен электрохимиялық тұрақтылығын басқаруда тұндыру температурасы мен аралық қабат конфигурациясының шешуші рөл атқаратынын көрсетеді.

Түйін сөздер: литий-ионды аккумуляторлар, SnO_2, Ti/Pt аралық қабаты, радиожиілікті магнетронды шашырау, қыздыру, электрохимиялық көрсеткіштер.

Влияние условий осаждения на электрохимические характеристики тонких пленок SnO_2 в качестве анодов для литий-ионных аккумуляторов

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Аннотация. Тонкоплёночные аноды SnO_2 были изготовлены методом высокочастотного магнетронного (ВЧ) распыления металлической мишени Sn на подложки из нержавеющей стали с покрытием Ti/Pt. Осаждение проводилось при комнатной температуре (RT) и при 300 °C в атмосфере Ar/O_2 с последующим отжигом на воздухе при 620 °C. Структура, морфология и электрохимические характеристики плёнок были систематически исследованы. Анализ рентгеновской дифракции (XRD) подтвердил формирование чистой фазы тетрагонального рутила SnO_2 без обнаружения вторичных фаз. Методом сканирующей электронной микроскопии (SEM) выявлены значительные изменения размера зёрен и однородности поверхности в зависимости от температуры осаждения и термической обработки. Электрохимические испытания в монеточных ячейках CR2032 продемонстрировали характерный многостадийный механизм литирования SnO_2, включающий реакции конверсии и сплавообразования. Несмотря на то, что отжиг способствовал повышению кристалличности и снижению поляризации, плёнки, осаждённые при комнатной температуре, показали более высокую долговременную циклическую стабильность по сравнению с образцами, полученными при повышенной температуре. Полученные результаты подчёркивают ключевую роль температуры осаждения и конфигурации подслоя в контроле структурной целостности и электрохимической стабильности тонкоплёночных анодов SnO_2 для литий-ионных аккумуляторов.

Ключевые слова: литий-ионные аккумуляторы, SnO_2, межслой Ti/Pt, радиочастотное магнетронное распыление, отжиг, электрохимические характеристики.