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Chemically prepared La_2Se_3 nanocubes thin film for supercapacitor application





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ABSTRACT

Lanthanum selenide (La₂Se₃) nanocubes thin film is prepared via successive ionic layer adsorption and reaction (SILAR) method and utilized for energy storage application. The prepared La₂Se₃ thin film is characterized by X-ray diffraction, field emission scanning electron microscopy and contact angle measurement techniques for structural, surface morphological and wettability studies, respectively. Energy dispersive X-ray microanalysis (EDAX) is performed in order to obtain the elemental composition of the thin film. The La₂Se₃ film electrode shows a maximum specific capacitance of 363 F g⁻¹ in a 0.8 M LiClO₄/PC electrolyte at a scan rate of 5 mV s⁻¹ within 1.3 V/SCE potential range. The specific capacitive retention of 83 % of La₂Se₃ film electrode is obtained over 1000 cyclic voltammetry cycles. The predominant performance, such as high energy (80 Wh kg⁻¹) and power density (2.5 kW kg⁻¹), indicates that La₂Se₃ film electrode facilitates fast ion diffusion during redox processes.

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1. Introduction

The development of cost-effective, eco-friendly, fresh and good performance energy storage system are needed for modern society and as a result under consideration to increasing demands required

* Corresponding author. *E-mail address*: l_chandrakant@yahoo.com (C.D. Lokhande). electrical energy storage devices [1]. The energy storage devices including, batteries, supercapacitors, fuel cells, etc. are the essential part of the clean energy. A latest kind of alternative energy storage device, supercapacitor have superior advantages such as, high rate capability, good cyclic life span, superior reversibility, dynamics of charge–discharge propagation, and low maintenance cost [2]. Supercapacitors or electrochemical capacitors are employed for energy storage devices to construct the capacitances in several orders of magnitude larger than ordinary capacitors [3]. According to the charge storage mechanism, supercapacitor electrode materials are divided into two categories. The carbon based materials with high surface area and long cycle life appear under electrical double-layer capacitors (EDLCs) have storage capacity practically lower than that of batteries [4]. The metal oxides, metal chalcogenides, and conducting polymers with excellent redoxactivity and prominent electrochemical properties show the pseudocapacitive behavior with high specific capacitance compared with EDLCs [5].

Specifically, transition metal sulfides with multiple oxidation states are used as pseudocapacitive electrode materials [6-8]. Similarly, few metal selenide materials are also tested to supercapacitor applications [9-11]. Three-dimensional (3-D) GeSe₂ synthesized via a thermal evaporation method displayed a specific capacitance of 300 Fg^{-1} in flexible all-solid-state supercapacitors [9]. Two-dimensional (2-D) nano disks (NDs) like SnSe and nano sheets (NSs) like SnSe₂ synthesized via a phase controlled method showed maximum specific capacitance of 168 and 228 Fg^{-1} , respectively [10]. The nanoflakes like La₂Se₃ electrode exhibited the maximum specific capacitance of 332 Fg^{-1} in aqueous electrolyte, synthesized via chemical bath deposition method [11]. The supercapacitive performance of electrode material could be enhanced by tuning the working potential window $(E = CV^2/2)$. The rare earth chalcogenides have attracted great attention due to their interesting electrochemical properties such as wide potential range in supercapacitor applications. These strategies for the choice of lanthanum selenide provide a promising way with higher power and energy densities.

The aqueous electrolytes provide limited potential range to typically ~1 V, thereby limiting the achievable energy and power [12]. Hence, the supercapacitive performance of La₂Se₃ films is carried out in non-aqueous (propylene carbonate) electrolyte with 1.3 V/ SCE potential range. The non-aqueous electrolytes have attracted great interest as alternative to aqueous electrolytes as their low vitriolicity, stable electrochemical properties and wide potential window [13,14]. The effect of scan rate, charge/discharge current and number of stability cycles are studied by cyclic voltammetry (CV) and galvanostatic charge discharge (GCD) techniques, respectively using eight channeled battery cycler. The frequency dependence supercapacitive behavior of La₂Se₃ electrode is studied using electrochemical impedance spectroscopy (EIS) technique.

2. Experimental details

La₂Se₃ thin film on stainless steel substrate is carried out by SILAR method at room temperature (300 K). Analytical reagent grade lanthanum chloride (LaCl₃), selenium (Se) powder, anhydrous sodium sulfate (Na₂S) and acetic acid (C₂H₄O₂) were used in the deposition of La₂Se₃ thin film. For the deposition of La₂Se₃ film, 0.05 M LaCl₃ complexed with $C_2H_4O_2$ by adjusting the $pH \sim 3$ and used as cationic precursor solution, while sodium selenosulphate (Na₂SeSO₃) is used as anionic precursor solution. 0.1 M solution of Na₂SeSO₃ was made by dissolving 2 g Se powder and 20 g Na₂S in 100 ml of double distilled water with constant stirring for 10 h at 353 K [15]. The solution was sealed and kept overnight, the selenium separates out, which is filtered. The freshly prepared 0.1 M Na₂SeSO₃ solution was used as anionic precursor with pH equal to 10 ± 0.1 . The stainless steel substrate was immersed in a cationic precursor solution for 40 s to adsorb complexed lanthanum ion (La³⁺) on the substrate. The substrate was rinsed in double distilled water for 20 s to remove loosely bound species. Then the substrate was immersed in anionic precursor solution (Na₂SeSO₃) for 20 s. Again, the substrate was rinsed with double distilled water for 20 s to remove excess La2Se3 or unreacted species. Thus, one SILAR deposition cycle of La₂Se₃ film was completed. By repeating number of SILAR deposition cycles the thickness of film increases and gets a good quality thin film. The terminal thickness of 372 nm (density = 6.45 g/cm^3) for La₂Se₃ thin film was found for 80 SILAR deposition cycles. After 80 SILAR deposition cycles, decrease in film thickness attributed to the formation of outer porous layer, resulting the film peels off from the substrate [16]. The prepared films were annealed in air furnace at 473 K for 20 min and used further characterizations.

3. Results and discussion

3.1. Thin film formation and reaction mechanism

The synthesis process for La₂Se₃ thin film formation is schematically illustrated in Fig. 1. The cationic precursor solution of as, LaCl₃ is complexed with $C_2H_4O_2$. So formed lanthanum complex $[La(C_2H_4O_2)]^{3+}$ is adsorbed on the substrate surface.

The anionic precursor solution contains Na_2SeSO_3 . In alkaline medium, hydrolysis of sodium selenosulphate gives Se^{2-} ions as [17],

$$Na_2SeSO_3 + OH^- \leftrightarrow Na_2SO_4 + HSe^-$$
(1)

$$HSe^- + OH^- \leftrightarrow H_2O + Se^{2-}$$
 (2)

Finally, $[La(C_2H_4O_2)]^{3+}$ ions react with Se²⁻ ions in the reaction bath and La₂Se₃ film formation takes place as,

$$2[La(C_2H_4O_2)]^{3+} + 3Se^- \rightarrow La_2Se_3 + 2C_2H_4O_2$$
(3)

The film deposited on the stainless substrate is uniform and well adherent. The loading mass of La₂Se₃ thin film is determined to be \sim 0.24 mg cm⁻² (thickness with respective of the mass of electrode material) by gravimetric method.

3.2. Characterization techniques

The prepared thin film is characterized for structural and phase identification by X-ray diffraction (XRD) using Bruker AXS D8 advance model with Cu K α (λ = 0.154 nm) in the 2 θ range of 20–70°. The surface morphological and elemental analysis is carried out using FE-SEM (SIGMA, Carl Zeiss) technique. The wettability test was conducted for contact angle measurement (propylene carbonate-electrolyte) using Rame-Hart NRL CA Goniometer. The electrochemical performance of La₂Se₃ film electrode is studied by cyclic voltammetry (CV) and galvanostatic charge–discharge (GCD) technique using electrochemical instrument interface, automatic battery cycler (WB CS3000). Impedance analysis of La₂Se₃ film electrode is carried out by electrochemical impedance spectroscopy (EIS) using electrochemical workstation (ZIVE SP5)) to characterize the interface quality of the electrode and electrolyte.

3.3. Structural studies

The XRD pattern of La₂Se₃ film deposited on stainless steel substrate is shown in Fig. 2(A). The diffraction pattern show peaks at 28.08°, 31.02°, 37.43°, 47.19° and 67.70° corresponding to (220), (310), (321), (332) and (541) planes, respectively which matches well with JCPDS card-00-019-0659. The XRD pattern shows the polycrystalline nature of La₂Se₃ film with cubic crystal phase. Bagde et al. [18] has reported similar kind of crystal structure for La₂Se₃ material synthesized by a spray pyrolysis method. The peaks observed at 36.41° and 39.45° are assigned to LaSe₂ structure with (22–1) and (311) planes, respectively (JCPDS card: 01-075-2270). The asterisk '*' marked peaks indicate the contribution of stainless steel substrate in XRD pattern. The crystallite size of La₂Se₃ film material for (321) plane is found to be 84 nm. Download English Version:

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