PHASE STABILITY AND TRANSFORMATION OF ENERGY STORAGE MATERIALS

Novel Hydrothermally Synthesized Strontium Telluride Nanoballs as an Efficient Electrocatalyst for Oxygen Evolution Reaction

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The development of extremely efficient, low-cost, and stable electrocatalysts is required for the sluggish oxygen evolution reaction. Strontium telluride multi-skinned nano balls deposited on glassy carbon (SrTe/GC) were fabricated using the hydrothermal technique and were characterized via different analytical instruments. The fabricated SrTe/GC nanoballs underwent electro-chemical measurement, achieving a low overpotential of 268 mV at 10 mA/cm² with a smaller Tafel slope of 25 mV/dec. The fabricated material also exhibited excellent stability for 24 h with no decline in current density. Hence, this study shows that strontium telluride could function as an extraordinarily efficient and stable electrocatalyst in future energy conversion applications.

INTRODUCTION

Fossil fuels now account for the vast majority of the world’s energy, and these energy carriers must be phased out due to their rapid depletion, and environmental contamination.1 To compete with fossil fuels, scientists are exploring new renewable energy sources which must be environment friendly and have huge resources as well.2–4 Water electrolysis is one method of obtaining energy. It has the ability to store energy for an extremely long period of time. When molecular hydrogen is created, it is stored in an exterior tank. The water molecules are oxidised by the anode, producing protons, electrons, and molecular oxygen (OER). The protons flow through the anode’s electrolyte, while the cathode absorbs the electrons that pass through the external circuit, resulting in the emission of hydrogen molecules (HER). OER requires more effort because it moves slower and has more steps. There aren’t many OER catalysts because of metal dissolution, high oxidation in acidic conditions, and severe corrosion, and the ones that are accessible require noble metals like Ru and Ir. Water electrolysis in an acidic electrolyte, on the other hand, is a better approach to produce hydrogen. Because it is an alkaline media, research has concentrated on developing catalysts that are both effective and inexpensive. SrTe is an alkaline earth chalcogenide and one of the difficult strontium cogenides to comprehend. under 0.18, 0.142, and 0.12 Mbar pressures.

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environmental impact, hydrogen (H₂) is often promoted as a potential option to meet the growing global demand for clean and renewable energy resources.⁵–⁹ There are various renewable energy sources like wind,¹⁰ electrochemical water splitting, geothermal,¹¹ and solar power¹² for producing energy. Among many others, electrochemical water splitting has proven to be a viable choice to get sustainable, clean energy.¹³–¹⁵

For water splitting, the practical results for electrolytic polarization-induced overpotentials are sometimes substantially different from theoretical expectations.¹⁶,¹⁷ To overcome this issue, the precious metal electrocatalysts like IrO₂/RuO₂ for OER and Pt-based materials for HER are used, while large-scale applications of the precious metals are limited due to their high costs, lack of availability, and monotonous activity.¹⁸–²⁰ Sluggish OER electrocatalysts based on transition-metal compounds have been extensively explored to address the shortcomings of noble-metal-based catalysts. To increase the electrochemical performance, metal chalcogenides-based electrode materials have recently been investigated²¹ and outperformed other catalysts in the water oxidation process due to their porous nature.²²–²⁴ There has been ongoing research into metal-based materials such as oxides, hydroxides, phosphides, nitrides, and carbides.²⁵–²⁹ Transition metal chalcogenides, such as copper (Cu),²⁶ nickel (Ni),²⁷ manganese (Mn),²⁸ cobalt (Co),²⁹ molybdenum (Mo),³⁰ iron (Fe),³¹ and zinc (Zn),³² with different morphologies, have been investigated for electrochemical water splitting applications. A good electrocatalyst must have several characteristics, such as effective charge transfer, lower activation energy, surface active sites, and higher electric conductivity.³³,³⁴ The alkali metal chalcogenides have all such characteristics and their morphology can be changed with the reaction conditions to convert them into porous polyhedrons, hollow structures, nanocubes, nanorods, and nanoballs.⁴⁶,⁴⁷ Among all, 3-dimensional nanoballs have the increased surface area, improved electrolyte penetration, and also have ideal exposed crystal facets for OER [⁴⁸, ⁴⁹].

Hence, inspired by the effective architectural design, alkali metal chalcogenides have been rarely investigated for sluggish oxygen evolution reactions. SrTe can be considered an excellent candidate as an electrocatalyst for electrochemical water splitting due to its excellent conductivity, tunable morphology, high surface area with large number of active sites at the surface, and wide range of electronic properties.³⁹–⁴¹ Consequently, the investigation of SrTe in the bottleneck OER process is important. This study presents the fabrication of strontium telluride via a one-step hydrothermal route. After that, the fabricated material was deposited on the glassy carbon electrode and then employed for electrochemical measurements such as electrochemical performances.

**EXPERIMENTAL SECTION**

**Materials**

All the analytical-grade chemicals were used as such, without any further treatment, to fabricate the strontium telluride. Deionized water was used in the whole experiment, and the following chemicals were utilized: tellurium powder (Te, Merck, > 99.9%), strontium nitrate [Sr(NO₃)₂·6H₂O, Sigma Aldrich, > 99.9%], hydrazine monohydrate, (N₂H₄·H₂O, Panreac, > 80%), ethyl alcohol (Merck, 99.8%), and potassium hydroxide (KOH, Analar, 99%).

**Preparation of SrTe**

Ordinary conditions and a significantly simple setup were employed for one-step hydrothermal synthesis to produce SrTe nanoballs. The 0.01 M (0.255 g) strontium nitrate hexahydrate and 0.01 M (0.102 g) tellurium powder were mixed in 50 ml deionized water, followed by addition of 5.0 ml of 7.0 M KOH solution. After that, the mixture was homogenized for 2 h with the addition of 2.0 ml of hydrazine monohydrate under vigorous stirring. The resultant mixture was then placed in a 100-ml Teflon-sealed stainless-steel autoclave, and heated at 200°C for 12 h. To separate precipitates, the resultant mixture was centrifuged, washed with DI water and (C₂H₂OH) ethanol subsequently and dried out in an oven at 70°C overnight, and the obtained SrTe powder was stored for characterization purposes. A simple hydrothermal procedure for manufacturing the SrTe/GC nanoballs is shown in Fig. 1.

**Characterization**

The structural analysis was done by using powder x-ray diffraction (Bruker-D8 Advance diffractometer) employing Cu/Kz radiation having wavelength λ = 1.5406 Å, with a scanning rate of 5° per min, and operated at 40 kV/35 mA. Scanning electron microscopy (SEM) was used to reveal the morphology of the fabricated material (Quanta 200-FEG SEM). For this purpose, a small amount of catalyst was mixed in ethanol and sonicated for 10 min. After that, a drop of this dispersed catalyst was put on the conducting side of the FTO and allowed to dry at room temperature before SEM analysis. A transmission electron microscope [JEOL (JEM-2100) TEM] was used to analyze the deep internal morphology of the synthesized product. A drop of dispersed catalyst was pasted on a copper grid which was later used for the TEM analysis. An x-ray photoelectron spectroscopy (XPS- ESCALAB 250) analysis was used to analyze the sample’s composition (Thermo, Waltham, USA).

**Electrochemical Setup**

A Teflon-covered Pyrex glass cell was employed via a three-electrode electrochemical cell to perform
all electrochemical parameters in basic (1.0 M KOH) condition. The three-electrode configuration was maintained by using platinum wire as a counter electrode, platinum as counter electrode, and the SrTe treated on glassy carbon electrode as a working electrode connected with a computer-controlled PGSTAT AUTOLAB-204. The working electrode was formed by making a slurry from 5 mg of the fabricated material dissolved in 100 μl of deionized water. After that, the thick paste was deposited on the surface of the glassy carbon via the drop cast method. Before any measurements, the electrochemical cell was cleaned thoroughly with HNO₃:HCl (1:3), ultrapure water, and acetone subsequently, and then dried at 80 °C for around 30 min. The platinum wire was also washed using 10% nitric acid solution, and all the electrochemical tests were repeated 5 times to avoid any type of error during the experiments. As specified in Eq. 1, all potential measurements were taken using a reversible hydrogen electrode (RHE).

$$E_{\text{RHE}} = E_{\text{Ag/AgCl}} + E_{\text{o}} + (0.059 \times \text{pH})$$ (1)

To compensate for their 50% iR-compensation, the cyclic and linear sweep voltammetry were scanned at 5 mV/s. To learn about the catalytic and kinetic efficacy, the following equation was used to determine the Tafel slope.

$$\eta = x + 2.303 \frac{RT}{nF} \log j$$ (2)

where the number of electrons involved is denoted by n, the Faraday constant is F, the current density is j, and the slope is 2.303 RT/nF. Using the cyclic voltammetry (CV) curve in the non-faradic zone, the double-layer capacitance of the as-prepared working electrode was determined at a scan rate of 10, 20, and 30 mV/s. To calculate the electrochemical surface area (ECSA) of the synthesized material, the double-layer capacitance was divided by the specific capacitance, which is 0.04 mF cm⁻² considered for flat electrode using Eq. 3.

$$\text{ECSA} = \frac{C_{dl}}{C_s}$$ (3)

Electrochemical impedance (EIS) spectroscopy was used to measure the charge transfer resistances across an electrode-electrolyte interface in the frequency range of 0.1–100,000 Hz. For this purpose, NOVA 2.1 software and a simple Randles circuit were used to calculate the Rs and Rct from the Metrohm Autolab-204 electrochemical workstation. Each cyclic voltammetry curve was IR adjusted. To account for potential values, the following equation was used:

$$E_{\text{actual}} = E_{\text{experimental}} - \text{IR}$$ (4)

RESULTS AND DISCUSSION

Structural Analysis

The crystal structure, orientation, and phase purity of the SrTe powder were confirmed by the
XRD investigation. Figure 2 depicts the XRD pattern of the synthesized sample used to identify the crystalline phases and was well-matched with Joint Committee of Powder Diffraction (JCPDS) card No. 03–065–5700 (space group: Pm-3m and space group number: 221). Strontium telluride diffraction peaks positioned at \(2\theta = 24.1^\circ, 34.5^\circ, 42.3^\circ, 49.2^\circ, 55.4^\circ, 61.1^\circ, 72.1^\circ, \) and \(77.3^\circ\) indexed to hkl (100), (110), (111), (200), (210), (211), (220), and (221), respectively, corresponding to the cubic structure, and confirm its successful synthesis.

**Morphological Analysis**

Scanning electron microscopy (SEM) was used to reveal the shape of the SrTe electrocatalyst. The resultant SEM micrographs of SrTe display the multi-skinned nano balls in Fig. 3a and b at low and high magnifications. As observed from the SEM micrographs, the multi-skinned nanoballs have an average diameter of 82 nm, and the designed multi-skinned architecture was responsible for a large active sites number, and keep intact for the ease of ion and electron transportation across the interface of the electrode and electrolyte. To gain deep insight into the morphology, transmission electron microscopy (TEM) was used, and the resultant micrograph is shown in Fig. 3c, which shows consistency with the SEM micrograph.

**X-ray Photoelectron Microscopy**

X-ray photoelectron spectroscopy (XPS) was used to determine the precise structure and chemical makeup of the SrTe/GC surface, as well as the various oxidation states of the metals. Figure 4a shows the XPS survey spectrum of SrTe, and elements present in the sample, i.e., Sr, Te, C, and O. The dominant peaks of Sr3d and Te3d, present at 133 eV and 582.4 eV, respectively, are shown in Fig. 4b–d, confirming the oxidation states of +2 and −2, respectively, validating the successful synthesis of SrTe. The signals from C at 278 eV were used for the calibration, while some background oxygen (O) signals are detected at 532 eV, due to the surface
adsorbed O, as a result of the exposure to air/moisture on the surface of the catalyst.

**Electrochemical Characterization**

The OER experiment was carried out using computer-controlled three-electrode configurations (PGSTAT, AUTOLAB-204) at a sweeping rate of 5 mV/s in an electrolyte containing 1.0 M KOH catalyzed by SrTe nanoballs deposited on glassy carbon (GC) electrodes. The fabricated material was employed for electrochemical measurements like CV, LSV, EIS, and CA. The resultant cyclic and linear voltammograms are depicted in Fig. 5a and b. The SrTe/GC has a highly integrated peak area, implying that many Sr-charged species are involved in the generation of oxygen gas. The anodic peak value of the Sr redox reaction for SrTe/GC at 1.48 V versus RHE and overpotential of 268 mV at 10 mA/cm² current density through the CV and LSV curves are shown in Fig. 5a and b. The CV curve for SrTe/GC exhibits enhanced current density and decreased overpotential due to the formation of hydroxotelluride and oxyhydroxotelluride in alkaline media. The sharp and steep peak confirms the active strontium ions as a result of intermediate hydroxotelluride and peroxide production to speed up the sluggish oxygen evolution reaction. Table I shows a comparison of the electrochemical activity of SrTe/GC nanoballs with other catalysts. The exceptional OER activity of simple metal telluride makes it superior to bimetallic chalcogenides.

Electrochemical impedance is an important technique for measuring intrinsic electrocatalytic activity. The EIS test shown in Fig. 5c for the SrTe/GC electrocatalyst exhibits low charge transfer resistance ($R_{ct}$) and solution resistance ($R_s$) of 3.5 and 3.2 Ω, respectively. Also, the semicircle in a low-frequency region demonstrates small resistance to charge transfer and high electrical conduction for smooth OER performance.

For a better understanding of the kinetics of the electrochemical water oxidation, the Tafel slope was computed from the linear part of the plot between the logarithmic current density versus overpotential calculated from CV as shown in Fig. 5d. The OER kinetics of the SrTe/GC nanoballs appear to be superior due to the smaller Tafel slope of 25 mV. The value of the Tafel slope in the present work is much smaller or comparable to previously reported metal telluride-based electrodes (Table I). Simple electron transfer and rate-determining steps have long been associated with the Tafel slope. According to the Tafel slope, there is a 4-electron transfer mechanism that occurs for the OER rate-determining step, as given in Eqs. 5, 6, 7 and 8.36

$$\text{SrTe} + \text{OH}^- \leftrightarrow \text{SrTe} - \text{OH} + e^- \quad (5)$$

$$\text{SrTe} - \text{OH} + \text{OH}^- \leftrightarrow \text{SrTe} - \text{O} + \text{H}_2\text{O} + e^- \quad (6)$$

$$\text{SrTe} - \text{O} + \text{OH}^- \leftrightarrow \text{SrTeOOH} + e^- \quad (7)$$

![Fig. 4. (a) Survey spectrum of SrTe, (b–e) XPS spectra of Sr3d, Te3d, C1s, and O1s.](image)
CV cycles at different scan rates (10, 20, and 30 mV/s) were performed in a narrow voltage range in the non-faradic region, as shown in Fig. 6a. Double-layer capacitance is used to calculate the ECSA, using Eq. 3. A double-layer capacitance ($C_{dl}$) value of 0.009 F was obtained, as shown in Fig. 6b. By considering the specific capacitance of 0.04 mF/cm$^2$, the ECSA value of the hierarchical nanoballs was found to be 225 cm$^2$. Hence, the SrTe/GC nanoball increases mass transport kinetics due to increased contact area between the electrode and the electrolyte, resulting in faster charge transfer, and also the fast transport kinetics across the electrode-electrolyte, as was confirmed via EIS interface due to the high conductivity during the oxygen evolution process.

Using repeated CV scans under the same electrochemical conditions as for CV and EIS, the long-term survival of the electrocatalyst was examined using cyclic voltammetry and chronoamperometry. In a 1.0-M KOH solution, the CV scans were recorded for up to 1000 cycles at a scan rate of 50 mV/s. The first and 1000th CV cycles provide almost the same electrochemical signal with a little bit of

$$\text{SrTe} - \text{OOH} + \text{OH}^- \leftrightarrow \text{SrTe} + \text{O}_2 + \text{H}_2\text{O} + e^-$$

(8)
change in the current density, as shown in Fig. 7a. The stability of the fabricated sample was also confirmed via chronoamperometry at an applied potential of 0.8 V. The I-t curve resulting from CA remains stable up to 13 h as depicted in Fig. 7b. A slight decrease in current density was also observed in an alkaline environment in 1.0-M KOH. Hence, the nanostructures are suitable for large-scale commercial applications because they can penetrate and eliminate gas bubbles from the electrode edge. To confirm the stability of the fabricated material, the XRD analysis was also performed after the stability test, as shown in Fig. 7c, confirming that no phase change occurred as the peaks remain the same as those seen before the stability test.
CONCLUSION

A simple hydrothermal technique was used to synthesize the multi-skinned nanosized strontium telluride (SrTe) with ball-like morphology. The fabricated material had an enhanced electrochemical surface area of 225 cm², a lower onset potential of 1.48 V, and a lower overpotential of 268 mV to attain a current density of 10 mA/cm². It also required a Tafel slope of 25 mV/dec and confirmed the 4-electron transfer mechanism. The improved electrochemical performance was due to the high crystallinity and nanoball-shaped morphology with a lower value of charge transfer resistance. This research looks at the viability of synthesizing metallic telluride with a certain shape, high activity, and stability at a high current density to increase OER performance and produce high purity H₂ at a large scale.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest. No potential competing interest was reported by the authors.

REFERENCES

1. M. U. Nisa, S. Manzoor, A. G. Abid, N. Tamam, M. Abdullah, M. Najam-Ul-Haq, M. Al-Buriahi, Z. Alrowaili, Z. M. Mahmoud, and M. N. Ashiq, Fuel 321, 124086 (2022).
2. K. Handayani, Y. Krozer, and T. Filatova, Energy Policy 127, 134 (2019).
3. C. Liu, F. Li, L.-P. Ma, and H.-M. Cheng, Adv. Mater. 22, E28–E62 (2010).
4. H. Nong, L. Jiang, N. Yang, C. Yang, X. Zhu, Y. Jiang, X. Shen, C. Li, H. Tran, C. Spörri, R. Mom, J. Timoshenko, A. Knop-Gericke, G. Zichittella, and Y. Krozer, ChemNanoMat 6, 336 (2020).
5. I. Hassan, H. S. Ramadan, M. A. Saleh, and D. Hissel, Renew Sus. Energ. Rev. 149, 113131 (2021).
6. E. Mayousse, F. Maillard, F. Fouda-Onana, O. Sicardy, and N. Guillet, Int. J. Hydrog. Energy 7, 26410 (2019).
7. J. Lee, I. Kim, and S. Park, Int. J. Hydrog. Energy 33, 138769 (2020).
8. I. Hassan, H. S. Ramadan, M. A. Saleh, and D. Hissel, Renew Sus. Energ. Rev. 149, 113131 (2021).
9. Z. Abdin, A. Zafaranloo, A. Rafiee, W. Mérida, W. Łipiński, and K. R. Khalipour, Renew. Sust. Energ. Rev. 120, 109620 (2020).
10. L. Jiang, N. Yang, C. Yang, X. Zhu, Y. Jiang, X. Chen, C. Li, and Q. Sun, Appl. Catal. B: Environ. 280, 118780 (2020).
11. M. Ghazvini, M. Sadeghzadeh, M. H. Ahmadi, S. Moosavi, and F. Pourfayaz, Int. J. Energy Res. 45, 7823 (2020).
12. B. J. Ng, L. K. Putri, Y. X. Kong, Y. W. Teh, P. Pasbakhsh, and S. P. Chai, AdV. Sci. 7, 1900171 (2020).
13. X. Hu, X. Tian, Y.-W. Lin, and Z. Wang, RSC Adv. 9, 31563 (2019).

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