

Hierarchical porous NiCo_2Se_4 grown on carbon nanotube film via a hyperbranched polymer template for flexible asymmetric supercapacitors

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Abstract Lightweight carbon nanotubes (CNTs) have garnered significant interest in the realm of flexible electronics; however, achieving both high capacitance and high energy density simultaneously remains a formidable challenge. The application of epoxy-terminated hyperbranched polymer (EHP) is presented as a reactive template for the *in-situ* synthesis of porous NiCo_2Se_4 on a pristine CNT film. The as-prepared porous NiCo_2Se_4 exhibits a core-shell nanostructure and demonstrates a larger surface area and superior electrochemical properties compared to NiCo_2Se_4 synthesized without the CNT film. When employed as an electrode material, the CNT@ NiCo_2Se_4 film achieves a high specific volume capacitance of $281.7 \text{ F} \cdot \text{cm}^{-3}$. The flexible symmetric supercapacitor (SC) fabricated from the CNT@ NiCo_2Se_4 film exhibits a high specific volume capacitance ($23.3 \text{ F} \cdot \text{cm}^{-3}$), retains 95.6% capacitance retention after 5000 charge-discharge cycles, displays excellent bending resilience, and achieves a high energy density ($3.2 \text{ mWh} \cdot \text{cm}^{-3}$). Moreover, the device sustains stable performance across diverse bending conditions, underscoring its promise for flexible and wearable electronic applications. Within this system, the CNT film functions as a flexible, conductive current collector, while the porous NiCo_2Se_4 provides ample electroactive sites, collectively facilitating outstanding electrochemical performance. The reaction mechanism underlying the use of epoxy-terminated hyperbranched polymer as a reactive template in the preparation of porous NiCo_2Se_4 was elucidated through FT-IR, XRD, and TG analyses. The findings reveal that the EHP-templated approach effectively facilitates the formation of a porous NiCo_2Se_4 nanonetworks on CNT films, which improves electrical conductivity, creates abundant active sites, and improves ion/electron transport. Consequently, this strategy significantly enhances the overall electrochemical performance and energy-storage capability of the assembled flexible asymmetric supercapacitor.

Keywords hyperbranched polymers; carbon nanotubes film; flexible asymmetric supercapacitor; template method; NiCo_2Se_4 nanonetwork

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超支化聚合物模板法改性碳纳米管薄膜原位构筑 多孔 NiCo_2Se_4 柔性非对称超级电容器

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摘要 轻质碳纳米管薄膜在柔性电子领域备受关注,但如何同时提升其高电容与能量密度仍面临挑战.报道了一种以环氧端基超支化聚合物作为反应性模板,在原始碳纳米管薄膜上原位制备多孔NiCo₂Se₄的方法.所得多孔NiCo₂Se₄具有核壳纳米结构,与未使用碳纳米管薄膜制备的NiCo₂Se₄相比,展现出更高的比表面积和电化学性能.作为电极材料时,CNT@NiCo₂Se₄薄膜表现出281.7 F·cm⁻³的高体积比电容.以其组装的柔性非对称超级电容器具有高体积电容(23.3 F·cm⁻³)、5000次充放电循环后95.6%的电容保持率以及良好的弯曲性能,同时实现了3.2 mWh·cm⁻³的高能量密度.此外,该器件在不同弯曲状态下仍能保持稳定性能,显示出其在柔性和可穿戴电子器件中的应用潜力.在该体系中,碳纳米管薄膜既作为柔性集流体,又提供了良好的支撑,多孔NiCo₂Se₄则提供了丰富的活性位点,从而实现优异的电化学性能.利用环氧端基超支化聚合物作为反应性模板制备多孔NiCo₂Se₄的反应机制并通过FT-IR、XRD和TG进行了分析.结果表明:EHP模板策略能够有效促进CNT薄膜上多孔NiCo₂Se₄纳米网络结构的形成,从而提高电导率、提供丰富的活性位点,并改善离子/电子传输,进而显著提升柔性非对称超级电容器的整体电化学性能和储能能力.

关键词 超支化聚合物;碳纳米管薄膜;柔性非对称超级电容器;模板法;NiCo₂Se₄纳米网络

Amid growing global attention on the development of sustainable energy, rechargeable batteries and supercapacitors (SCs) have been broadly investigated and deemed as promising highly efficient energy storage systems^[1-2]. SCs have gained great interests as a power source for their high power density, fast charge-discharge rate, long discharge time and high cycle life. Although SCs can deliver high power density, their practical applications are restricted by the low energy density. According to the equation $E=1/2 CV^2$, two promising approaches to address this issue are to synthesize high capacitive electrode materials and construct asymmetric SCs (ASCs)^[3]. ASC can combine a battery-type faradaic electrode and a capacitance-type electrode to increase the output voltage^[4]. Thus, developing appropriate electrode materials has become the essential part to boost the energy densities.

Recently, electrochemically active transition metal selenides have attracted considerable interest as promising electrode candidates for supercapacitors owing to their superior electrical conductivity and high theoretical capacitance. NiCo₂Se₄ is considered to be a promising electrode material. Compared with a single nickel and cobalt selenide, NiCo₂Se₄ has high conductivity and abundant redox reaction sites which can provide better electrochemical performance^[5]. Moreover, rational design of electrode structures, such as three-dimensional core-shell architectures, can further enhance electrochemical performance by providing abundant active sites and facilitating ion/

electron transport^[6]. Rational design of morphology and/or structure provides one of the most feasible ways to create high performance SCs. Currently, the hydrothermal method is widely employed for the preparation of NiCo₂Se₄^[7-9]. For instance, Akbar *et al*^[10] successfully synthesized NiCo₂Se₄ microspheres via a two-step hydrothermal approach. The electrode obtained in this work showed a remarkable specific capacitance of 2112.30 F·g⁻¹ along with excellent cycling stability. Furthermore, an asymmetric device assembled with FeS₂ demonstrated exceptional overall performance, realizing a specific capacitance of 221.30 F·g⁻¹ and a high energy density of 78.68 Wh·kg⁻¹. Compared with the above processes, a new strategy is proposed to construct NiCo₂Se₄ using epoxy-terminated hyperbranched polymer (EHP) as both a template and electrode additive. Hyperbranched polymers are superior soft template materials because of their low-viscosity and large number of terminal functional groups^[11], which offer active sites to nucleate and grow nanomaterials. Such a method could significantly enhance the kinetic performance and long-term cycling stability. Our group have previously prepared Cu₉S₅, NiCo₂O₄ and CuS using an amino-ended hyperbranched polyamide and a hydroxyl hyperbranched polyamide as template, respectively^[12-14].

Herein, we synthesized unique hierarchical NiCo₂Se₄ nanonets on carbon nanotube (CNT) film by using EHP as a template. The resultant CNT@NiCo₂Se₄ composite exhibited high electrochemical performance

and was employed as the positive electrode in asymmetric devices. As a result, the designed ASC based on CNT@NiCo₂Se₄//CNT@PPy yielded an extended voltage window up to 1.5 V. At the power density of 101.35 mW·cm⁻³, the highest energy density of 7.90 mWh·cm⁻³ can be reached, and the energy density of 5.41 mWh·cm⁻³ was still maintained at the maximum power density of 1013.51 mWh·cm⁻³. After 6000 cycles, the ASC device still had 85.98% of the original capacitance.

1 Experimental section

1.1 Materials and instruments

All chemicals were of analytical reagent grade and used without further purification. Sodium selenate (Na₂SeO₄), hydrazine hydrate (N₂H₄·H₂O), and potassium hydroxide (KOH) were purchased from Sinopharm Chemical Reagent Co., Ltd. Multi-walled carbon nanotubes (MWCNTs) were supplied by Aladdin Industrial Corporation. Deionized water was used throughout the experiment.

The experiments were carried out using the following instruments: an electronic analytical balance (model BSAA224S, Beijing Sartorius Instruments Co., Ltd.), an electrochemical workstation (model CHI760E, Shanghai Chenhua Instrument Co., Ltd.), a coin cell testing system (model CT-4008T-5V50mA, Shenzhen Neware Electronics Co., Ltd.), a vacuum drying oven (model DZF-6020, Shanghai Sopo Instrument Co., Ltd.), a high-speed centrifuge (model TG16-WS, Changsha Xiangzhi Centrifuge Instrument Co., Ltd.), and a thermostatic heating magnetic stirrer (model DF-101S, Wuhan Keer Instrument Equipment Co., Ltd.).

1.2 Preparation of NiCo₂Se₄ positive electrode

NiCo₂Se₄ nanonets were prepared by a two-step process. The NiCo₂O₄ was obtained according to our previous report^[15]. Then, NiCo₂O₄ was converted to NiCo₂Se₄ by hydrothermal treatment in a selenide solution. Typically, Na₂SeO₄ (0.2 g) and hydrazine

hydrate (10 mL) were first mixed under vigorous stirring and then sealed in a Teflon-lined stainless-steel autoclave, followed by heating at 120 °C for 10 h. Finally, the converted sample was washed with distilled water and dried at 50 °C under vacuum.

1.3 Preparation of PPy negative electrode

Electrochemical characterization was performed in a three-electrode setup consisting of a CNT film as the working electrode, an Ag/AgCl electrode as the reference, and a platinum foil as the counter electrode. The PPy deposited with a constant voltage of 0.8 V in deionized water (57 mL) solution including NaClO₄ and pyrrole (3 mL) monomer for 2 h.

1.4 Fabrication of asymmetric symmetric SCs

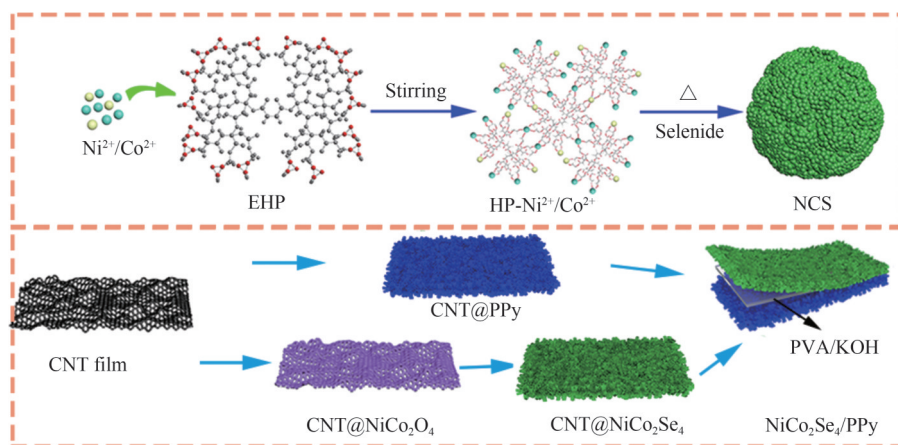
The NiCo₂Se₄ nanocomposite film and PPy was coated with PVA/KOH gel electrolyte several times to ensure uniformity and dried at 50 °C for 1 h. The identical coated NiCo₂Se₄ and PPy were placed face to face to form an ASC for electrochemical characterization.

2 Results and discussion

2.1 Fabrication and structural characterization of electrode materials

The fabrication process of the NiCo₂Se₄ and assembly of the ASC are illustrated in Fig.1. In detail, the EHP was used as a reactive template, metal ions (Ni²⁺, Co²⁺) attack the oxygen atoms on the terminal epoxy groups. The oxygen atoms after ring opening form a coordination covalent bond with the metal ions, which provides a directed point to prepare a metal salt precursor with EHP. Verified in previous work, EHP acts as the template and nucleophilic reagent, and its terminal epoxy functional group becomes a more active site for nucleation and growth of nanomaterials.

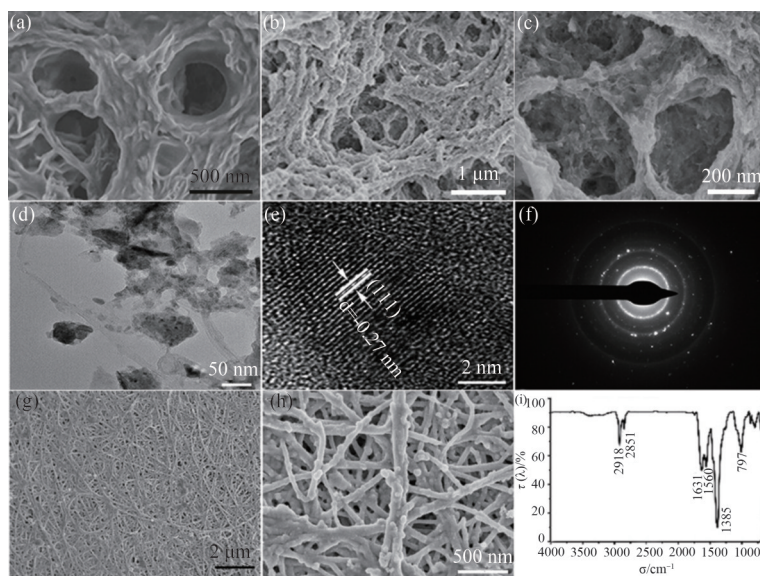
Scanning Electron Microscope images (Fig.2(a)-(c)) reveal that CNT@NiCo₂Se₄ maintains a three-dimensional porous network structure after selenization, in which CNTs are uniformly covered by NiCo₂Se₄ nanoflakes, forming a core-shell architecture. This structure facilitates electrolyte accessibility and enhances

Fig. 1 Fabrication of NiCo_2Se_4 and the ASC based on NiCo_2Se_4 //PPy图1 NiCo_2Se_4 的制备及基于 NiCo_2Se_4 //PPy 的非对称超级电容器

electrochemical performance. Transmission Electron Microscope observations (Fig. 2 (d)) further confirm that NiCo_2Se_4 is coated on the CNT surface, consistent with the SEM results. The HRTEM image (Fig. 2 (e)) shows a lattice spacing of 0.278 nm, corresponding to the (101) plane of NiCo_2Se_4 , while the SAED pattern (Fig. 2 (f)) indicates its polycrystalline nature. In addition, CNT@PPy exhibits a dense yet porous network structure (Fig. 2 (g), (h)), where PPy is uniformly coated on individual CNTs, providing efficient ion transport pathways. The FT-IR (Fig. 2 (i)) shows characteristic peaks corresponding to N—H, C=C, and C—N vibrations, confirming the successful

formation of conductive PPy.

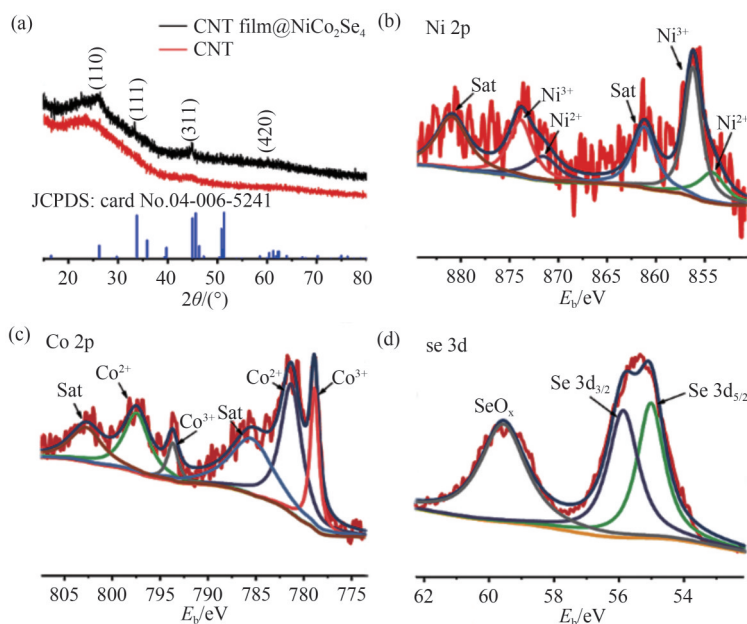
The sample was analyzed by X-Ray Diffraction (Fig. 3 (a)), the main diffraction peaks in the spectrum correspond to the NiCo_2Se_4 (JCPDS 04-006-5241). X-ray photoelectron spectroscopy (XPS) was conducted to examine the chemical bonding states of the surface elements in NiCo_2Se_4 . The electron-bonding energies of Ni 2p_{3/2} and Ni 2p_{1/2} at 856.2 and 873.8 eV correspond to Ni²⁺ cations^[16] (Fig. 3 (b)). The Co 2p spectrum (Fig. 3 (c)) exhibits characteristic peaks corresponding to Co²⁺ and Co³⁺ along with satellite peaks, confirming the coexistence of multiple valence states^[17-18]. The Se 3d spectrum (Fig. 3 (d)) shows two characteristic



(a) NiCo_2O_4 @CNT SEM image; (b), (c) NiCo_2O_4 @CNT SEM images; (d) NiCo_2O_4 @CNT TEM image; (e), (f) HRTEM image and selected area electron diffraction (SAED) pattern; (g), (h) CNT@PPy SEM images; (i) infrared spectrum of CNT@PPy at 4000~1500 cm^{-1}

Fig. 2 Morphology and microstructure of electrode materials

图2 电极材料的形貌与微观结构



(a) XRD pattern; (b) High-resolution XPS spectrum of Ni 2p; (c) High-resolution XPS spectrum of Co 2p; (d) High-resolution XPS spectrum of Se 3d

Fig. 3 Phase and surface chemical composition analysis of NiCo_2Se_4

图3 NiCo_2Se_4 的物相与表面化学组成分析

peaks assigned to $\text{Se } 3d_{5/2}$ and $\text{Se } 3d_{3/2}$, indicating the formation of metal-selenium bonds. These results further confirm the successful synthesis of NiCo_2Se_4 .

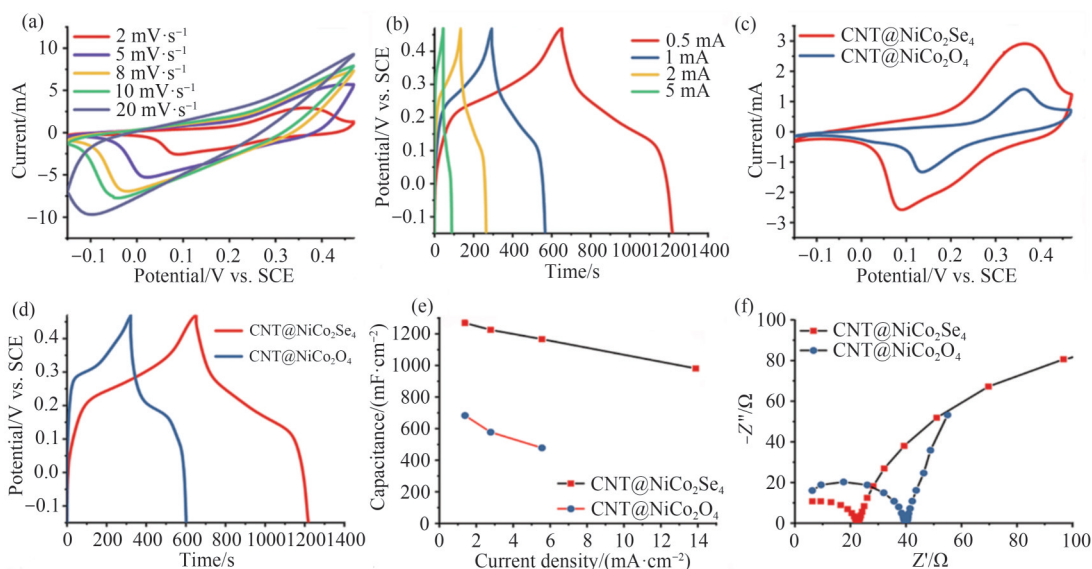
2.2 Electrochemical performance of $\text{CNT@NiCo}_2\text{Se}_4$ positive electrode

The electrochemical test of $\text{CNT@NiCo}_2\text{Se}_4$ was carried out with $3 \text{ mol} \cdot \text{L}^{-1}$ KOH aqueous solution as the electrolyte, and the voltage window was determined to be $-0.15 \sim 0.47 \text{ V}$. As shown in Fig. 4 (a), with the increasing sweep rate, the current response increases without any obvious change in the shape of the CV curves, indicating that $\text{CNT@NiCo}_2\text{Se}_4$ has better rate performance as an electrode material. A pair of redox peaks can be clearly observed at $2 \text{ mV} \cdot \text{s}^{-1}$, which corresponds to $\text{Co}^{2+}/\text{Co}^{3+}$ at 0.06 V , and corresponds to the conversion of $\text{Ni}^{2+}/\text{Ni}^{3+}$ at 0.35 V . Fig. 4 (b) shows the galvanostatic charge-discharge (GCD) curve of $\text{CNT@NiCo}_2\text{Se}_4$, which shows obvious charge-discharge platform due to its pseudo-capacitance characteristics. Meanwhile, all GCD curves at different currents appear to have a relatively symmetric shape with visible potential plateaus. Fig. 4 (c) is a comparison of $\text{CNT@NiCo}_2\text{Se}_4$ and $\text{CNT@NiCo}_2\text{O}_4$ under $10 \text{ mV} \cdot \text{s}^{-1}$. The CV curve of $\text{CNT@NiCo}_2\text{O}_4$ is completely

surrounded by $\text{CNT@NiCo}_2\text{Se}_4$ which shows that the performance of $\text{CNT@NiCo}_2\text{Se}_4$ has been significantly improved. Fig. 4 (d) is a GCD comparison of $\text{CNT@NiCo}_2\text{Se}_4$ and $\text{CNT@NiCo}_2\text{O}_4$ at $1 \text{ mA} \cdot \text{cm}^{-2}$. The discharge time of $\text{CNT@NiCo}_2\text{Se}_4$ is twice than that of NiCo_2O_4 . Fig. 4 (e) is a comparison of capacitance under different current densities of the two samples, which confirms that $\text{CNT@NiCo}_2\text{Se}_4$ delivers a higher area specific capacitance. Furthermore, the $\text{CNT@NiCo}_2\text{Se}_4$ delivers a lower impedance than $\text{CNT@NiCo}_2\text{O}_4$ (Fig. 4 (f)). In the Nyquist plot, the high-frequency semicircle represents the charge transfer resistance (R_{ct}), and the low-frequency slope reflects ion diffusion kinetics. The smaller semicircle of $\text{CNT@NiCo}_2\text{Se}_4$ confirms its superior conductivity and faster reaction kinetics via the EHP template method.

2.3 Electrochemical performance of CNT@PPy negative electrode

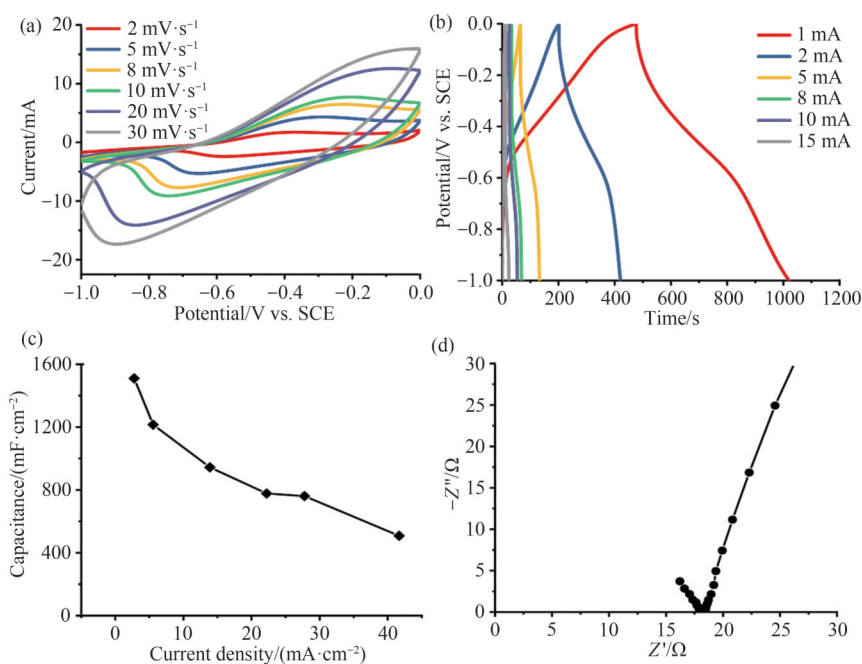
Fig. 5 (a) is the CV curve of CNT@PPy . Obvious redox peaks can be observed at low scan rates. With increasing the scan rates, there are no obvious changes in the curve, indicating that CNT@PPy has better rate performance. Fig. 5 (b) is the GCD curve of



(a) CV curves; (b) GCD curves, comparison of electrochemical performance of $\text{CNT@NiCo}_2\text{O}_4$ and $\text{CNT@NiCo}_2\text{Se}_4$; (c) CV curves measured at $10 \text{ mV} \cdot \text{s}^{-1}$; (d) GCD curves collected at $1 \text{ mA} \cdot \text{cm}^{-2}$; (e) capacitance at different discharge currents; (f) Nyquist plots

Fig. 4 Electrochemical characterization of $\text{CNT@NiCo}_2\text{Se}_4$ in a three-electrode configuration

图 4 $\text{CNT@NiCo}_2\text{Se}_4$ 在三电极系统中的电化学性能



(a) CV curves; (b) GCD curves; (c) capacitance at different current density; (d) Nyquist plot

Fig. 5 Electrochemical performance of CNT@PPy under a three-electrode configuration

图 5 CNT@PPy 在三电极系统中的电化学性能

the electrode under different currents. Obvious charge and discharge platforms can be observed, corresponding to the redox peaks. Fig. 5 (c) shows the specific capacitance calculated from the GCD curve. At a current density of $0.33 \text{ mA} \cdot \text{cm}^{-2}$, the electrode delivers an areal capacitance of $1526.38 \text{ mF} \cdot \text{cm}^{-2}$, reflecting its superior electrochemical storage capacity. Fig. 5 (d)

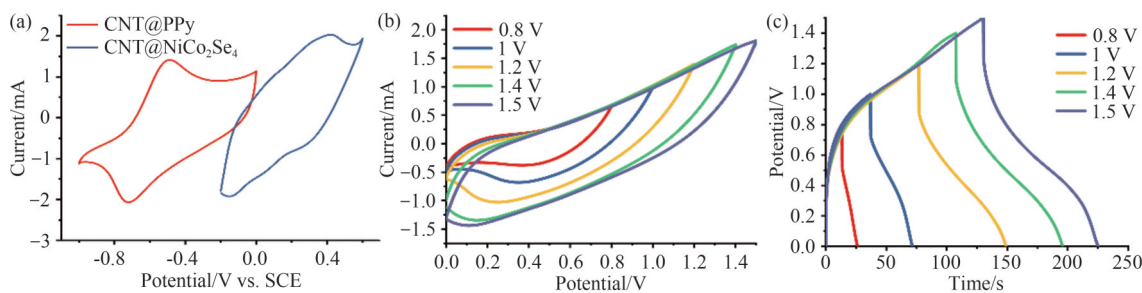
shows an impedance value of 18.2Ω , indicating low internal resistance. In addition, the slope is close to 1 in the low frequency region, indicating excellent capacitance performance.

2.4 Assembly and voltage window of the asymmetric supercapacitor

Based on the positive and negative materials that

have been successfully prepared, CNT@NiCo₂Se₄ and CNT@PPy were employed as the positive and negative electrodes, respectively, and an ASC device was assembled using KOH/PVA gel as the electrolyte. The mass ratio was optimized based on the charge balance principle ($q^+ = q^-$) using $q = C \times \Delta V \times m$. As shown in

Fig.6 (a), the ratio of CV area at 30 mV s⁻¹ is 1:1. Fig.6(b) and 6(c) show the CV and GCD curves of the ASC devices. As the voltage window increases, these CV and GCD curves still maintain similar shapes, indicating that 1.5 V is a stable operating voltage for devices.



(a) CV curves of CNT@NiCo₂Se₄ and CNT@PPy electrode, and the assembled asymmetric device at a scan rate of 10 mV·s⁻¹; (b) CV curves of the as-prepared device measured over the operating voltage windows from 0 to 1.5 V at a constant scan rate of 30 mV·s⁻¹; (c) GCD profiles at different operating voltage windows at a current of 0.5 mA

Fig. 6 Electrochemical performance tests of different electrodes and the asymmetric device

图6 不同电极及非对称器件的电化学性能测试

2.5 Electrochemical Performance of the Asymmetric Supercapacitor

As illustrated in Fig. 7 (a), the CV profiles recorded at various scan rates over a potential window of 0-1.5 V exhibit two distinct redox peaks, which can be attributed to reversible faradaic reactions, in agreement with the results shown in Fig. 7 (b). The ASC device delivers a high areal capacitance of approximately 48 F·cm⁻² at a current density of 0.2 mA·cm⁻², which retains about 38 F·cm⁻² even at a high current density of 500 mA·cm⁻², demonstrating excellent rate capability (Fig. 7(c)). The impedance spectrum (Fig. 7(d)) reveals a relatively low internal resistance (46.4 Ω), indicating efficient charge transfer in the ASC device. The According to the Ragone plot (Fig. 7(e)), when operated at a power density of 101.35 mW·cm⁻³, the device exhibits an energy density reaching 7.90 mWh·cm⁻³, and still maintains 5.41 mWh·cm⁻³ even when the power density increases to 1013.51 mW·cm⁻³, reflecting its superior energy-power characteristics. Furthermore, after undergoing 6000 charge-discharge cycles, the ASC still maintains 85.98% of its original capacitance, indicating reliable long-term cycling stability. (Fig. 7

(f)). The flexible ASC is evaluated under different deformation conditions and continuous deformation tests. It can be observed that the capacity remains almost unchanged at 0.2 mA (Fig. 7(g)). Due to the limited power of a single ASC, it is difficult to apply it alone in real life. Therefore, ASCs are connected in series or parallel to meet specific requirements. From Fig. 7(h), the CD curve of two ASCs in series shows a voltage range that is twice of a single device and the charge and discharge time is not much different from that of a single device. The parallel connection of two ASCs has the same voltage window as a single device (Fig. 7(i)), but the discharge time is about twice that of a single device. Thus, our solid-state ASC as a reliable and powerful energy storage system can be used potentially in various practical applications.

3 Conclusion

In summary, CNT@NiCo₂O₄ was successfully prepared on a carbon film using hyperbranched as a template, followed by a simple one-step selenization to prepare a CNT@NiCo₂Se₄ composite film, which exhibits higher specific capacitance and lower

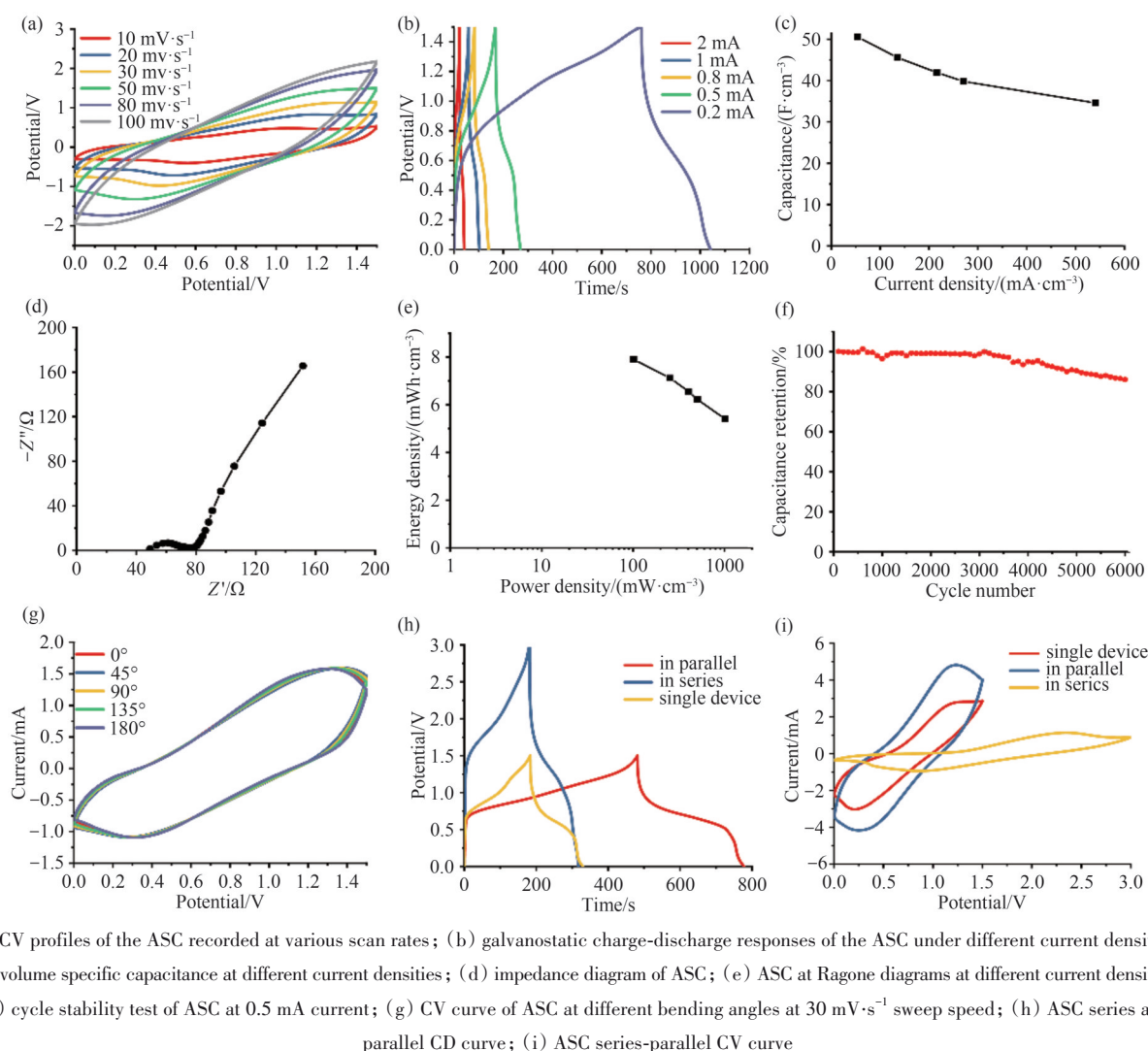


Fig. 7 Electrochemical performance characterization of the asymmetric supercapacitor (ASC) under diverse testing conditions

图 7 不对称超级电容器在不同测试条件下的电化学性能表征

impedance than CNT@NiCo₂O₄. The ASC device is assembled based on CNT@NiCo₂Se₄ positive and CNT@PPy negative electrode. The voltage window of the asymmetric device can be as high as 1.5 V, and the ASC delivers a high energy density of 7.8 F·cm⁻³. After 6000 charge and discharge cycles, it still exhibits a high capacitance retention of 85.98% at 0.5 mA.

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