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Graphene Composites for Lithium-ion Battery Anodes: A Review

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Abstract: New energy technologies are advancing fast, pushing up the demand for higher-energy-density lithium-ion batteries. Graphene composites stand out among anode modification strategies due to their good conductivity and flexibility. They have been applied to silicon, sulfide, and oxide anodes. This review covers recent progress in graphene composite anodes, looks at how graphene works in these systems, and discusses future directions.

Keywords: Graphene–anode composites; lithium-ion batteries; transition metal sulfides; silicon anodes; metal oxides.

1. Introduction

The theoretical capacity of graphite anodes is stuck at 372 mAh/g. That is not enough for today's energy storage needs. Lithium-ion batteries power phones, cars, and grid systems. They have good energy density and cycle life. But the anode is the weak link. To go further, we need better materials.

Silicon, transition metal sulfides, and metal oxides all beat graphite on theoretical capacity. Silicon tops out at 4200 mAh/g. SnS_2gives about 1136 mAh/g. VS_4reaches 1196 mAh/g. That is why these materials are getting so much interest.Tin-based sulfides like SnS_2offer about 1136 mAh/g. These high-capacity materials are not perfect. Conductivity is low, so electrons move slowly. And they swell—silicon by 300%, $GeO_2$by about 230%. That swelling cracks the electrode. Active material flakes off. Capacity fades fast.

Graphene is a two-dimensional atomic crystal made of carbon atoms with sp^2 hybridization. It has a honeycomb structure and is the basic building block of other carbon allotropes [1]. When stacked in three dimensions, it forms graphite. Rolled into one dimension, it becomes carbon nanotubes. Wrapped into zero dimensions, it becomes fullerenes [2]. Graphene is the thinnest known 2D material, with a layer spacing of only 0.335 nm [3]. Its C—C bond length is 1.42 Å, and the bond angle is 120°. In the 2D plane, each carbon atom forms a σ bond with its neighbors and contributes a π bond from the Pz orbital [4]. This unique band structure gives graphene excellent mechanical, thermal, electrical, and optical properties. It is considered the thinnest, hardest, and fastest electron-conducting material. Its carrier mobility reaches 200,000 $cm^2 \cdot V^{-1} \cdot s^{-1}$ at room temperature, more than ten times that of silicon. Its thermal conductivity is 5,300 $W \cdot m^{-1} \cdot K^{-1}$, far higher than copper (about 400 $W \cdot m^{-1} \cdot K^{-1}$). It also has a light absorption rate of 2.3%, a mechanical strength of 130 GPa (about 100 times that of steel), and a specific surface area of 2,630 $m^2 \cdot g^{-1}$ [5].

Thanks to its high conductivity, large surface area, good flexibility, and chemical stability, graphene offers an ideal solution for high-capacity anodes. Compositing high-capacity materials with graphene builds an efficient electron transport network. At the same time, graphene's flexible nature buffers volume expansion. Its 2D confinement effect also prevents nanoparticle agglomeration and keeps the electrode structure intact. Therefore, studying the use of graphene composites in lithium-ion battery anodes is important for developing next-generation high-energy-density batteries.

2. Graphene Preparation Methods

There are two main approaches to prepare graphene: top-down and bottom-up. In the top-down approach, graphite is exfoliated into graphene by chemical redox, exfoliation techniques (such as electrochemical, liquid-phase, or mechanical exfoliation), or arc discharge. The bottom-up approach uses carbon-containing precursors like gases, polymers, or aromatic hydrocarbons to synthesize graphene through chemical vapor deposition, epitaxial crystal growth, or other methods.

2.1. Top-Down Methods

2.1.1. Oxidation-Reduction Method

The redox method works in two steps. First, natural graphite is oxidized into graphene oxide (GO). Then, GO is reduced to obtain graphene. This method is low-cost and gives a high yield. Another advantage is that GO has many oxygen-containing groups on its surface. This makes it easy to combine with other materials or to modify further.

In the literature reviewed here, Xiao Bin et al. mixed GO with a tin-antimony precursor [6]. They then heated the mixture at 180°C for 10 hours in a hydrothermal reactor. The GO was reduced and coated onto the surface of SnS_2/Sb_2S_3. Tu Min et al. mixed SnS_2 with GO and heated it at 180°C for 2 hours to obtain SnS_2/RGO [7]. Liu Xinyue et al. first synthesized C@CoSe [8]. They then added GO and heated the mixture at 130°C for 8 hours. During this step, the GO was reduced to rGO. Chen Xiaoyi et al. used GO prepared by an improved Hummers method as the precursor. They heated it at 160°C for 24 hours to obtain VS_4@rGO [9].

He Fangfang et al. reduced an HSQ/GO precursor in a H_2/Ar atmosphere at 900–1050°C for 4 hours [10]. Zhang Lin et al. mixed $ZnMn_2O_4$ with GO and heat-treated the mixture at 350°C for 1 hour [11]. The GO was reduced to rGO. Wang Baode et al. mixed ZnS with GO and pre-reduced the mixture at 300°C. They then exposed it to microwave radiation three times to obtain ZnS/rGO [12].

2.1.2. Exfoliation Method

The exfoliation method separates graphene layers by overcoming the van der Waals force between graphite layers.

This is done using mechanical force, liquid shear, or electrochemical intercalation. In mechanical exfoliation, the ball milling method uses the impact and shear of high-speed milling balls to produce relatively well-ordered graphene. In liquid-phase exfoliation, graphite is dispersed in a surfactant or organic solvent, and ultrasonication or stirring exfoliates it into high-quality graphene. In electrochemical exfoliation, graphite acts as an electrode. An applied voltage drives electrolyte ions into the interlayer space, pushing the layers apart and separating them into graphene.

2.1.3. Thermal Decomposition Method (Pyrolysis)

Pyrolysis is a method for making graphene films. It works by decomposing carbon-containing precursors at high temperatures, usually between 600 and 1200°C. The precursors can be hydrocarbon gases like methane or acetylene, or polymers like polyacrylonitrile and phenolic resin. The carbon atoms are then deposited onto a substrate surface to form graphene films.

At high temperatures, the C-H bonds in hydrocarbon molecules break. The carbon atoms then rearrange into an sp^2 hybridized graphene lattice. This method produces a large amount of graphene with good crystallinity. It also does not need metal catalysts. However, it usually requires high temperatures above 800°C. Amorphous carbon impurities may also appear in the final product.

In battery anode composite research, Pan Shuhui et al. [13] pyrolyzed a precursor at 600°C for 2 hours under a nitrogen atmosphere. During this process, organic compounds in the precursor — such as polyvinylpyrrolidone and ammonium thioacetate — were carbonized. This formed a nitrogen-doped amorphous carbon layer. At the same time, FeS/SnS grains grew. Although this method did not directly produce graphene films, it used pyrolysis carbonization to build a carbon coating layer.

2.2. Bottom-Up Methods

2.2.1. Chemical Vapor Deposition (CVD)

Chemical vapor deposition (CVD) is a common method for making high-quality graphene. It uses carbon-containing gases like methane (CH_4), ethylene (C_2H_4), or acetylene (C_2H_2). These gases are mixed with hydrogen and argon in a tube furnace or a vacuum chamber.

The reaction happens on a metal substrate, such as copper or nickel foil, at high temperatures between 800 and 1050°C. The gases decompose on the metal surface, and carbon atoms deposit and self-assemble into continuous graphene films.

Here is a typical CVD process. First, copper foil is heated to 1000°C in argon and hydrogen to anneal it. Then, methane is introduced. Carbon atoms nucleate and grow on the copper surface. After the reaction, the system is cooled quickly. Finally, the copper substrate is removed by chemical etching, and the graphene is transferred to a target substrate.

CVD gives good control over the number of graphene layers, defect density, and grain size. That is why it is a main method for making large-area, high-quality graphene.

However, CVD has some drawbacks. The equipment is expensive, and the process conditions are strict. Also, transferring graphene from the metal substrate can introduce wrinkles and damage. These issues limit its direct use for making powder materials for battery anodes.

2.2.2. Epitaxial Growth Method

The epitaxial growth method uses silicon carbide (SiC) single crystals as the substrate. The process is carried out under ultra-high vacuum, around 10^{-9} to 10^{-10} Torr, and at very high temperatures, about 2180°C.

Under these conditions, silicon atoms leave the SiC surface first. The remaining carbon atoms then rearrange themselves into a graphene film.

The process usually has two steps. First, the SiC surface is etched with hydrogen at about 1000°C to make it atomically flat. Then, the material is graphitized at high temperatures. The number of graphene layers can be controlled by changing the heating time and temperature.

The graphene produced this way bonds strongly with the SiC substrate. It has high crystal quality and does not need to be transferred. This method is mainly used for high-frequency electronics, quantum computing, and other high-end applications.

3. Application of Graphene Composite Materials in Anodes for Lithium-Ion Batteries

3.1. Sulfide-Based Graphene Composites

3.1.1. Tin-Based Sulfide/Graphene Composites

Xiao Bin et al. prepared SnS_2/Sb_2S_3@G composites using a hydrothermal method. In this material system, graphene forms a multilayer coating around the SnS_2 particles [6]. This helps reduce volume expansion during cycling. Graphene and Sb_2S_3 also form a heterostructure together. This heterostructure improves the material's conductivity.

Electrochemical tests show that after 1200 cycles at a high current density of 2 A/g, the composite still delivers a specific discharge capacity of 521.7 mAh/g (Figure 1 [6]). The capacity shows no sign of decline. This demonstrates excellent cycle stability.

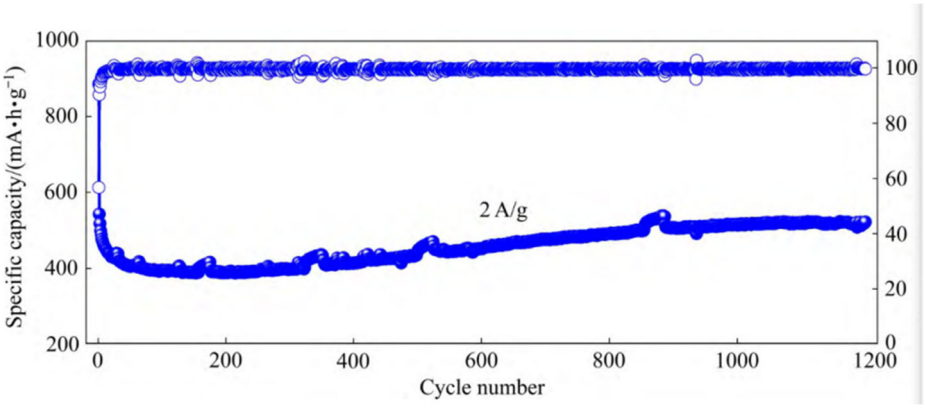

Figure 1. Cycling performance of SnS2/Sb2S3@G at current density of 2 A/g[6]

Tu Min et al. prepared SnS_2/RGO composites using a hydrothermal method. RGO has a high specific surface area [7]. This provides abundant active sites and also improves conductivity.

At 100 mA/g, the initial discharge capacity of SnS_2/RGO reached 2803 mAh/g (Figure 2 [7]). After 100 cycles, it still held 941 mAh/g. In comparison, pure SnS_2 delivered only 198 mAh/g after 100 cycles under the same conditions (Figure 2 [7]). This shows that adding RGO greatly improves the cycling stability of SnS_2.

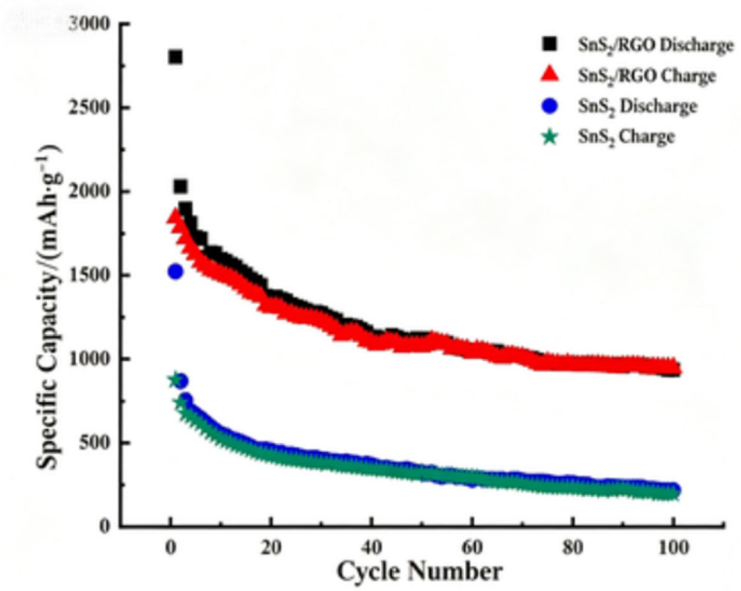

Figure 2. Cycle stability of SnS2 and SnS2/RGO at 100 mA/g[7]

Pan Shuhui et al. used ferric chloride and tin chloride as raw materials [13]. They prepared FeS/SnS@N-rGO composites through a solvothermal reaction followed by high-temperature pyrolysis.

The composites have a sandwich-like structure. The flaky rGO wraps around the FeS/SnS particles. This helps prevent volume expansion during charge and discharge.

At 0.1 A/g, the initial charge and discharge capacities reached 818.6 and 1430.0 mAh/g (Figure 3 [13]). After 100 cycles, the discharge capacity stayed at 594.5 mAh/g (Figure 3 [13]). These values are much higher than those of the sample without rGO.

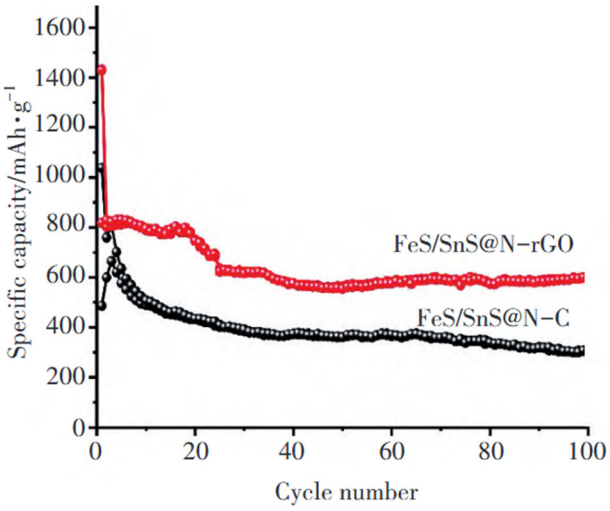

Figure 3. Cycling performance of FeS/SnS@N-rGO at 0.1 A·g^{-1}[13]

3.1.2. Other Metal Sulfide/Graphene Composites

Chen Xiaoyi et al. designed and synthesized VS_4@rGO composites [9]. In this material, VS_4 nanorods are tightly attached to graphene flakes. The graphene prevents the nanorods from clumping together into spheres. This frees up more active sites.

Rate performance tests gave the following results. At 0.2, 0.5, 1.0, 2.0, and 5.0 A/g, the average discharge capacities of VS_4@rGO were 983, 797, 666, 547, and 397 mAh/g (Figure 4(a) [9]).

The charge-discharge curves at different current densities all look similar. They have clear platforms. Even at high current densities, the battery still shows good platform behavior. This means the material can handle high currents well (Figure 4(b) [9]).

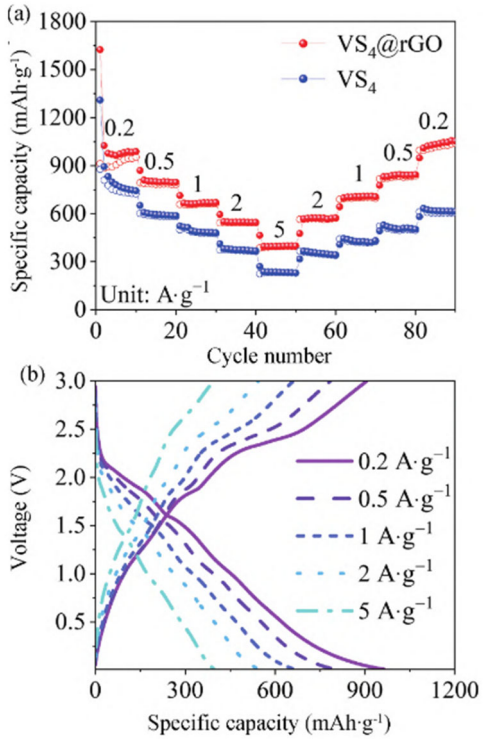

Figure 4. (a) Rate performance of LIBs with VS4 and VS4 @ rGO; (b) Charge and discharge curves of LIBs with VS4 @ rGO at different rates[9]

The VS_4@rGO electrode also showed good cycling performance. At 2 A/g, its initial discharge capacity was 784.9 mAh/g. After 800 cycles, it stayed at 565.8 mAh/g. The average capacity loss per cycle was only 0.035% (Figure 5(a) [9]).

At a higher current density of 5 A/g, the electrode ran for 3000 cycles. The capacity settled at 141.6 mAh/g. The average decay rate was only 0.019% per cycle (Figure 5(b) [9]).

For comparison, pure VS_4 electrodes performed much worse under the same conditions. This shows that adding graphene greatly improves the cycling stability of the material.

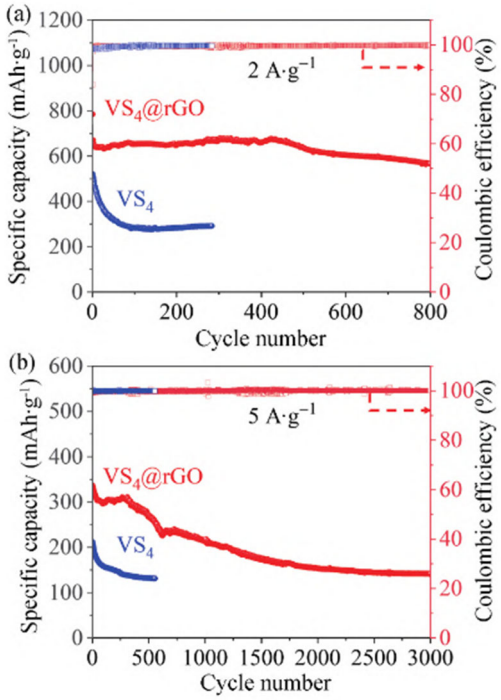

Figure 5. Cycling performance (left y - axis) and coulombic efficiency (right y-axis) of VS4 @ rGO and VS4 at (a) 2 A·g-1 and (b) 5 A·g-1[9]

Liu Xinyue et al. prepared porous C@CoSe/rGO composites using glucose and graphene as carbon sources [8]. The hydrothermal method was used.

The graphene and carbon layers work together as a dual

protection system. This improves the material's conductivity. It also reduces volume expansion and pulverization of the cobalt selenide particles. At the same time, it stops direct contact between cobalt selenide and the electrolyte.

At 200 mA/g, after 150 cycles, C@CoSe/rGO-2 delivered 751 mAh/g (Figure 6(a) [8]). At higher currents of 1000 and 2000 mA/g, after 150 cycles, it still gave 525 and 285 mAh/g (Figures 6(b) and 6(c) [8]).

Rate tests were also done. At 200, 400, 600, 1000, and 2000 mA/g, C@CoSe/rGO-2 had higher average capacities than the other two composites. When the current went back to 200 mA/g, the capacity returned to about 780 mAh/g. This is close to its initial capacity (Figure 6(d) [8]).

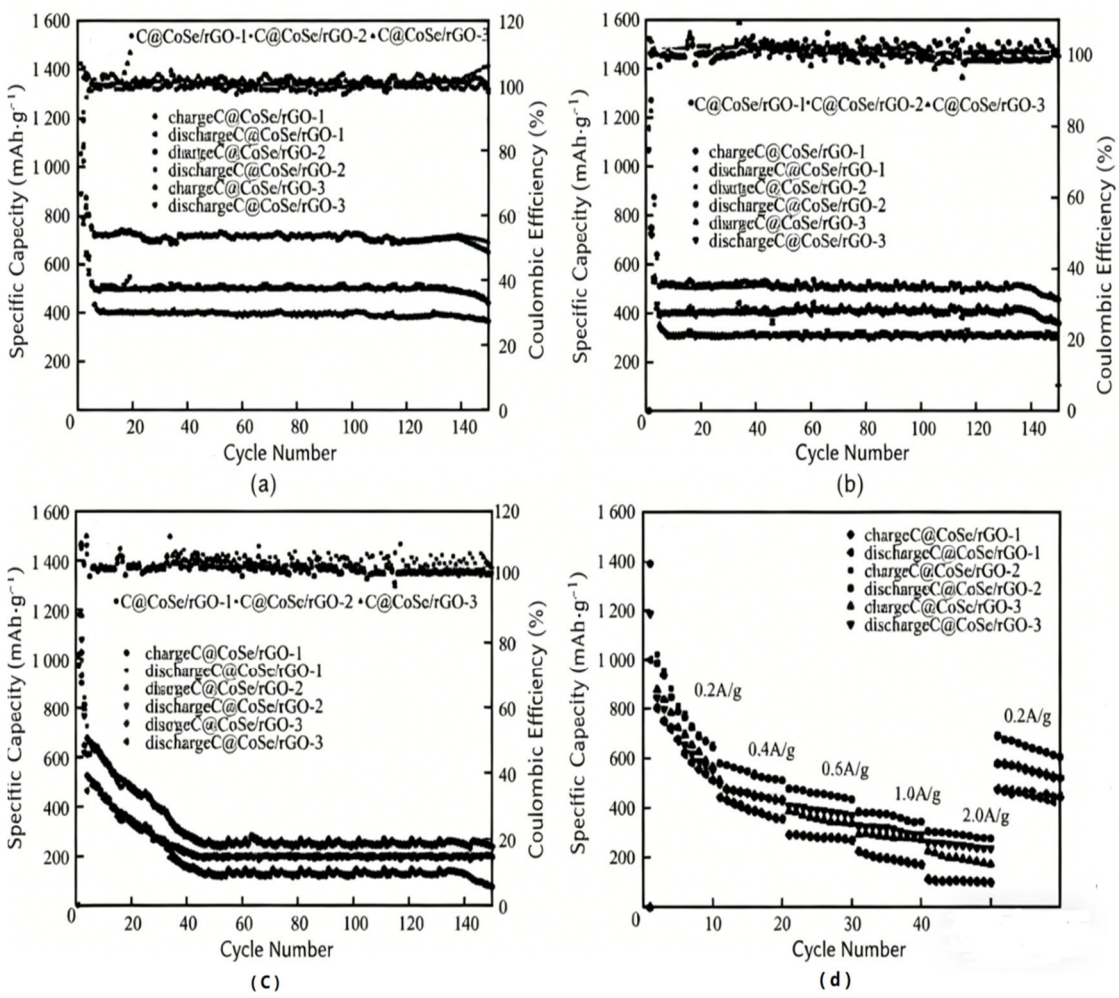

Figure 6. Comparison of cycling performance and rate capability of three composites[8]

Wang Baode et al. prepared ZnS/rGO composites using a microwave method [12]. They used zinc blende and graphene oxide as raw materials. They made two samples with different ZnS to GO mass ratios: 1:1 and 2:1. These were named ZnS/rGO-I and ZnS/rGO-II.

Electrochemical tests showed that the ZnS/rGO electrodes performed better than pure ZnS. They had higher initial discharge capacity, better first-cycle coulombic efficiency, and faster lithium ion diffusion.

For ZnS/rGO-I, the first-cycle coulombic efficiency was 56.09%. It rose to 89.78% in the second cycle (Table 1 [12]). At 0.5C, this electrode still delivered 111.1 mAh/g. When the current went back to 0.1C, the capacity recovered to 258.6 mAh/g. This shows a clear improvement in rate performance (Figure 7 [12]).

Table 1. Discharge specific capacity and coulombic efficiency of three electrode materials in the first three cycles

Parameter	ZnS	ZnS/rGO-I	ZnS/rGO-II
Initial discharge specific capacity / (mAh·g⁻¹)	614.16	618.60	484.61
Discharge specific capacity of the 2nd cycle / (mAh·g⁻¹)	277.72	364.70	240.64
Discharge specific capacity of the 3rd cycle / (mAh·g⁻¹)	154.24	334.10	167.61
Initial coulombic efficiency / %	46.34	56.09	52.32
Coulombic efficiency of the 2nd cycle / %	57.56	89.78	70.33

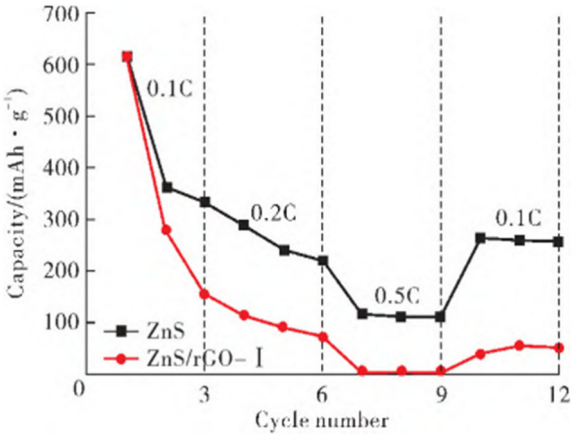

Figure 7. Rate performance of ZnS and ZnS/rGO-I electrodes [12]

3.2. Silicon-Based Graphene Composites

Li Cui'e et al. prepared Si/C composites by high-temperature pyrolysis [14]. They used orthogonal experiments to find the best process parameters.

The optimal conditions were: silicon to citric acid mass ratio of 1:9, pyrolysis temperature of 700°C, and pyrolysis time of 3 hours. Under these conditions, the Si/C composites had a flaky shape. The average particle size was 2.808 μm. The first charge capacity reached 3183 mAh/g.

Then they added graphene to the material. With 20% graphene, the first charge capacity went up to 3995 mAh/g (Figure 8 [14]). This is an increase of 812 mAh/g compared to the material without graphene. The electrochemical impedance also dropped from 271 Ω to 54 Ω (Figure 9 [14]).

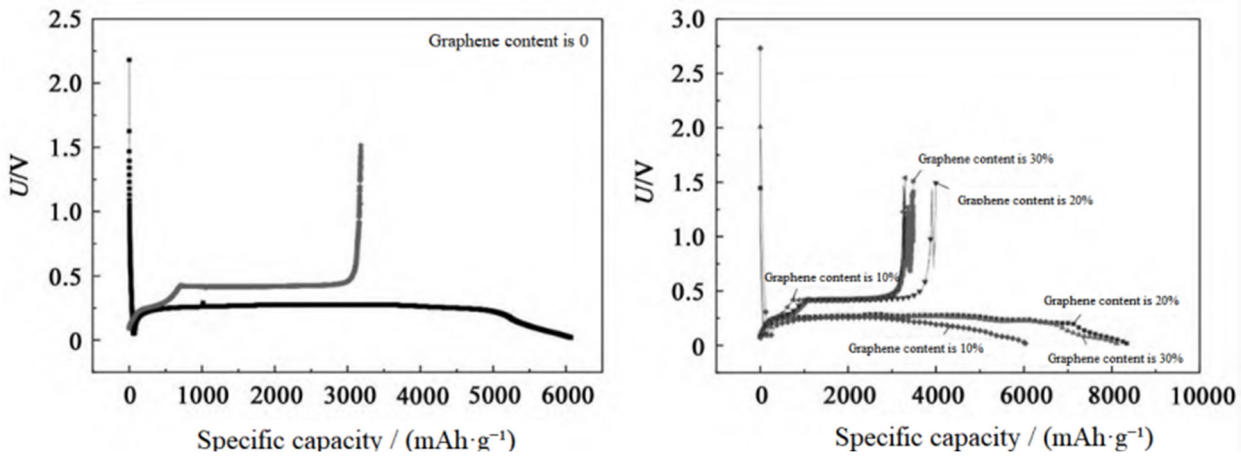

Figure 8. Constant current charge-discharge curves of Si/C composite[14]

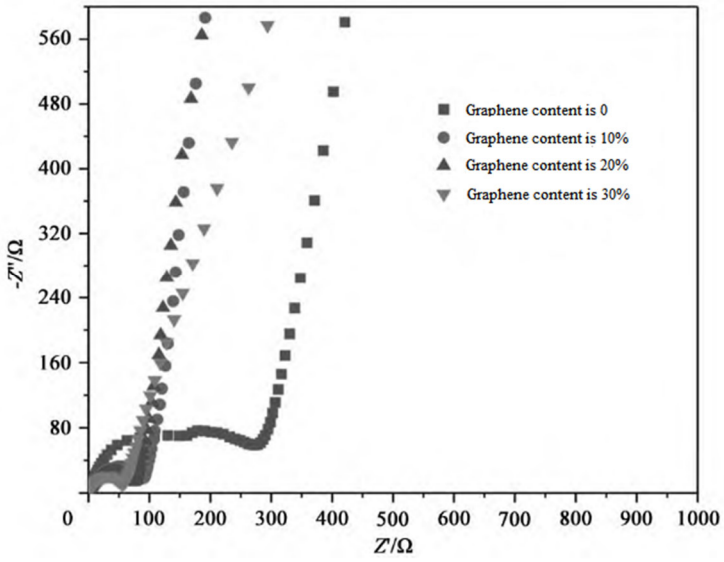

Figure 9. EIS curves of Si/C composite[14]

He Fangfang et al. prepared G/Si/SiO_x nanocomposites using a sol-gel method [10]. The G/Si/SiO_x-2 electrode was obtained by reduction at 1050°C. This electrode showed better performance.

Cyclic voltammetry tests gave the following results. The electrode had a smaller redox peak spacing. It also showed lower polarization. The peak shape remained stable during cycling (Figure 10 [10]).

Electrochemical impedance fitting results are shown in Table 2. The charge transfer resistance of G/Si/SiO_x-2 was 387.2 Ω. This is only 43.4% of that of the pure Si/SiO_x electrode.

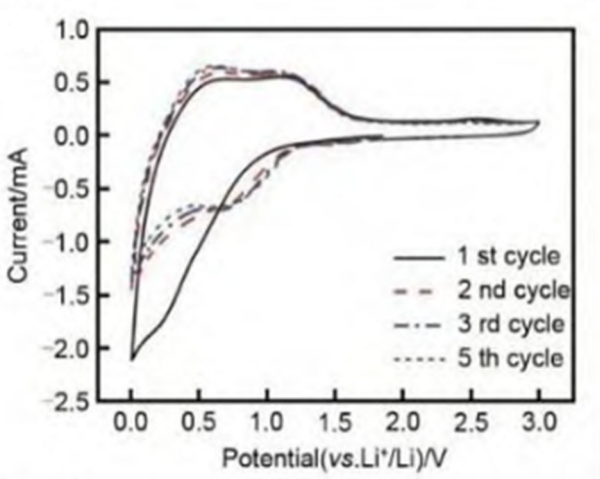

Figure 10. Cyclic voltammetry curve of G/Si/SiO_x-2[10]

Table 2. Electrochemical impedance fitting parameters

Electrode sample	Rs/Ω	Rct/Ω	CPE-T/(F·cm-2)	CPE-P
Si/SiOx	78.6	892.3	2.1×10^{-5}	0.82
G/Si/SiOx-1	65.3	516.7	3.5×10^{-5}	0.86
G/Si/SiOx-2	59.8	387.2	4.2×10^{-5}	0.88

3.3. Metal Oxide Graphene Composites

Zhang Lin et al. prepared $ZnMn_2O_4$ hollow cubes using a room-temperature microemulsion method [11]. The cubes have an edge length of about 200 nm. They are made of tightly connected nanoparticles, each about 30 to 50 nm in size.

Next, they mixed the $ZnMn_2O_4$ with graphene oxide. They then heat-treated the mixture at 350°C to get $ZnMn_2O_4$/rGO composites.

Thermogravimetric analysis shows that the rGO content in the composite is about 10.5% (Figure 11 [11]).

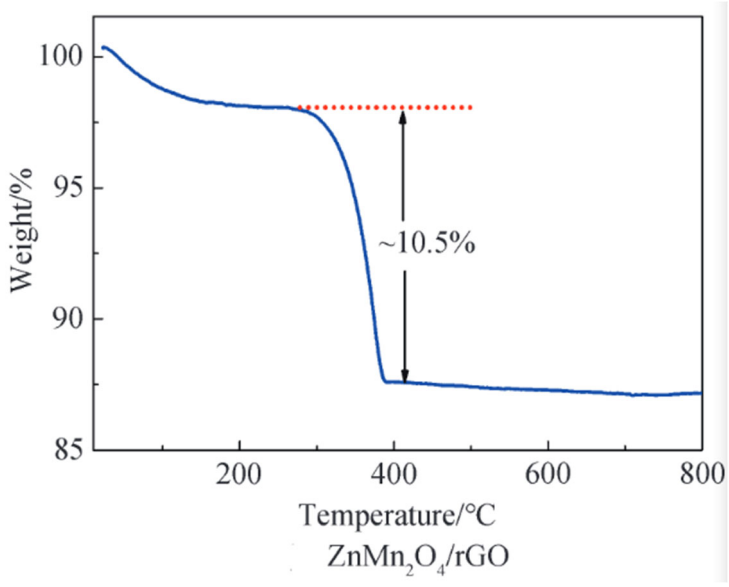

Figure 11. TG curve of ZnMn2O4/rGO[11]

This material has a unique hollow cubic structure. The hollow space helps buffer volume expansion during cycling. The rGO forms a three-dimensional electron transport network, which speeds up electron movement.

N_2 adsorption-desorption tests gave a specific surface area of 29.6 m^2/g and a total pore volume of 0.23 cm^3/g. The pore sizes are mainly in two ranges: 2–7 nm and 30–100 nm. The smaller pores come from gaps between particles. The larger

pores come from the hollow centers of the cubes (Figure 12 [11]).

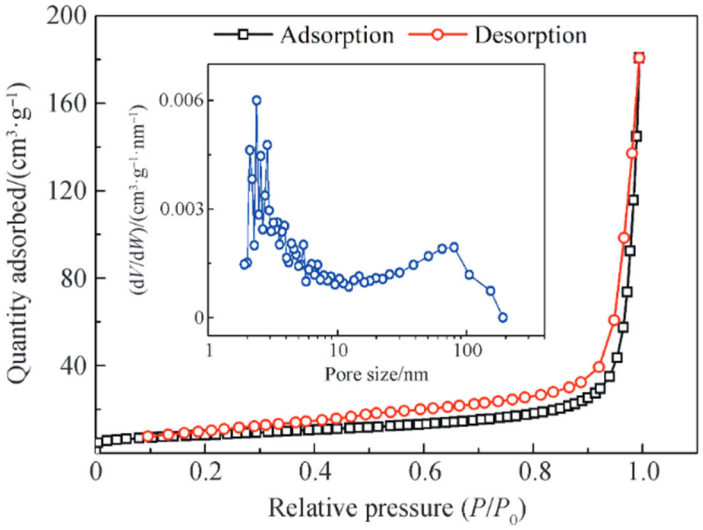

Figure 12. N2 adsorption-desorption isotherms with pore size distribution(inset)of ZnMn2O4/rGO[11]

Electrochemical tests gave the following results. At 0.1 A/g, $ZnMn_2O_4$/rGO delivered 1193 mAh/g. At 4 A/g, it delivered 620 mAh/g (Figure 13(a) [11]).

After 700 cycles at 1 A/g, the discharge capacity stayed at 806 mAh/g (Figure 13(b) [11]). Pure $ZnMn_2O_4$, under the same conditions, only kept 522 mAh/g. So adding rGO clearly improves cycling stability.

Electrochemical impedance spectroscopy gave a charge transfer resistance of 39.2 Ω for $ZnMn_2O_4$/rGO. For pure $ZnMn_2O_4$, it was 80.9 Ω (Figure 13(c) [11]). The lithium ion diffusion coefficient was 4.32×10^{-14} cm²/s for $ZnMn_2O_4$/rGO, compared to 2.34×10^{-14} cm²/s for the pure material (Figure 13(d) [11]).

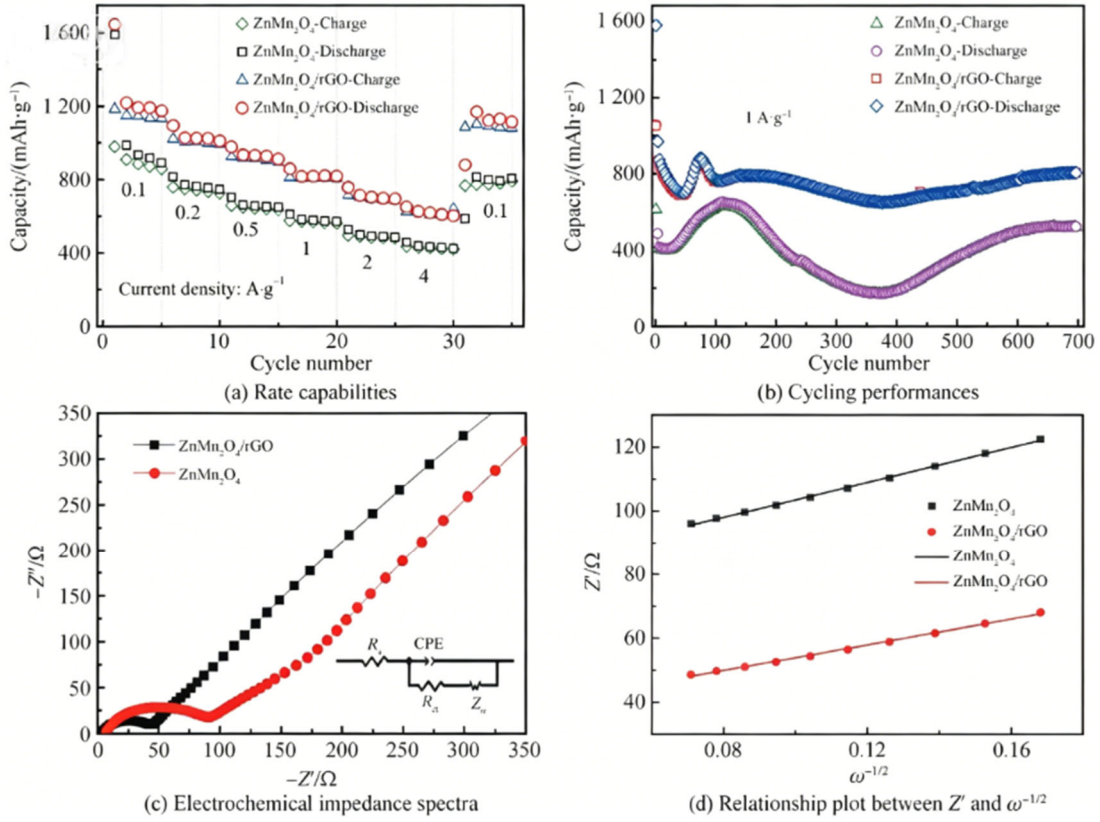

Figure 13. Lithium storage property tests of materials[11]

4. Discussion

Looking at the papers reviewed here, a few patterns stand out. Graphene helps all three types of anodes — sulfides, silicon, and metal oxides — last longer during cycling. The main reason is that graphene can take up the volume change. Rate performance depends a lot on how well the graphene network is built. A good graphene network lowers resistance

and speeds up lithium movement.

Among the three, silicon anodes give the highest capacity but also lose it fastest without enough graphene coating. Sulfide anodes, especially VS_4@rGO, can run for thousands of cycles with little decay. Metal oxide anodes like $ZnMn_2O_4$/rGO sit in the middle. Their hollow structure helps, so they balance capacity and stability well.

How graphene is made also matters. Most papers use hydrothermal reduction of GO. It works well and is easy to do. CVD and epitaxial growth make better graphene, but they cost too much and are hard to use for making powders. So for battery anodes, the hydrothermal method is the practical choice.

Putting graphene with high-capacity materials clearly works. But the interface between graphene and the active material could be better. And making graphene still costs too much.

5. Conclusion

Graphene solves two big problems of high-capacity anodes: low conductivity and large volume swings. It works in four ways. It builds a fast electron path. It bends to take up space changes. It keeps nanoparticles from clumping. And it helps form a stable SEI layer.

But there are still problems. Making graphene is expensive. The bond between graphene and the active material could be stronger. After many cycles, graphene sheets may stack back together.

Future work should try to cut costs, design better composites, and use simulations and real-time tests to see how the materials behave. Full-cell tests are also needed to check if these materials work in real batteries.

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