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过渡金属硫化物以及  $C_3N_4$  复合材料的可控  
制备及其光催化/电化学性能研究

Controlled Synthesis and Photocatalytical/Electrochemical  
Research of Transition Metal Sulfide and  $C_3N_4$  Composites

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**Controlled Synthesis and Photocatalytical/Electrochemical  
Research of Transition Metal Sulfide and C<sub>3</sub>N<sub>4</sub> Composites**



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## 摘要

化学掺杂（复合）和微观形貌调控是改变材料结构特征，进而改善材料性能两种主要手段。其中化学掺杂可以改变材料本身的价带和导带的位置，调整能带结构，并且掺杂的原子或复合的半导体化合物会与原材料产生一系列的互相激发反应，在材料表面或内部引起新的化学或物理反应，从而影响物质的性质；而微观形貌调控则是直接针对材料表面，控制合成不同的表面形貌，使其暴露出的活性位点发生改变，从而改变材料表面所带的基团及其比表面积，最终影响材料的光学、热学、电学、力学或化学特性。

在化学掺杂研究中，本文创新性的通过水热法合成了一种具有珊瑚状形貌的钴掺杂 ZnS。XRD, SEM, XPS 和 UV-Vis DRS 研究结果表明，在本文掺杂比例范围内，钴的掺入不影响形貌和结构，随着钴掺杂比例从 0 到 1，ZnS 的禁带宽度由 3.32 eV 减少至 2.65 eV，可见光吸收增加。另外，通过荧光测试发现此掺杂体系内的电子空穴复合得到了抑制。这两种改变均有利于光催化性能。在模拟自然光下，采用此掺杂体系进行光降解亚甲基蓝，效果证实适量的钴掺杂可以提高 ZnS 本身的可见光光催化效率。通过优化，得出 5.49 wt.% 的钴掺杂可得到最好的光降解亚甲基蓝效果。

在微观形貌调控方面，通过一步水热法合成了层数可调的具有层层自组装结构的  $\text{LaCO}_3\text{OH}$ 。研究发现，通过调控  $\text{CS}_2$  的加入量，可以制备出从 2D 到 3D 转变的具有不同层数的微观结构，且由于不同层状的  $\text{LaCO}_3\text{OH}$  表面缺陷不同，其荧光发射强度差异很大。此外，通过调节硫脲的含量得到了空心球和花型两种不同形貌的 CuS，这两种形貌的 CuS 表现出了不同的超级电容器性能，空心球 CuS 的比电容量可达花型 CuS 的近 3 倍。为了进一步提升空心球 CuS 的比电容量，还对其进行了碳量子点掺杂。研究结果表明，碳量子点能有效提高空心球 CuS 的比电容量，在碳量子点负载 5 wt.% 时为最佳。

除了硫化物和  $\text{LaCO}_3\text{OH}$ ，本文还研究了石墨烯型  $\text{C}_3\text{N}_4$  复合材料的电化学性能。通过一步煅烧法，将一定量的水合醋酸锌与硫脲粉末混合均匀并研磨处理，放至管式炉中 600℃ 高温煅烧，得到了 g- $\text{C}_3\text{N}_4$  与 ZnS 的复合物。XRD 和 XPS 分析结果验证了二者的成功复合，这表明 g- $\text{C}_3\text{N}_4$  与 ZnS 可以同时生成而不对彼此产生干扰。随后以泡沫镍作为集流体将此复合物做成电极，并考察其

在 6 M KOH 电解液中的赝电容器性能。通过优化, 得出当醋酸锌/硫脲质量比为 0.02 时其赝电容器性能最优, 此时复合体系不但有较高的比电容量, 还表现出优异的倍率性能和循环性能: 在 1 A/g 的充放电电流下, 比电容量为 497.1 F/g, 并且电压降仅为 0.12 V; 即使在较大的电流密度下, 如 10 A/g, 比电容量仍然有 345.9 F/g, 电容保持率为 69.5%。随后考察了此复合物体系的循环稳定性: 在 2 A/g 充放电 600 次后仍能保持 86% 的比电容量, 在 5 A/g 充放电 600 次后能保持 78% 的比电容量, 显示出较高的循环稳定性。通过替换此体系的集流体材料, 将泡沫镍改为不锈钢, 发现其优越的电容特性部分源于 Ni 参与了电化学反应。因此, 此超级电容器材料体系又可称之为 g-C<sub>3</sub>N<sub>4</sub>/ZnS/Ni 体系。

**关键词:** 硫化物; g-C<sub>3</sub>N<sub>4</sub>; 形貌控制; 光催化; 超级电容器

## Abstract

Chemical doping and morphology control are two ways proved to be efficient in improving the nano/micro-materials properties. The visible light absorption can be tuned with the band gap change led by heteroatom doping. Furthermore, new physical and chemical reactions with the pristine materials can be excited by the dopant elements or semiconductors, influencing various of material properties. Morphology control focuses on controlling the surface properties by changing the morphology of materials, various properties, such as optical, thermodynamical, electrical, mechanical or chemical properties of materials can be controlled due to the different exposed reactive sites with different morphologies.

In this thesis, Co-doped ZnS with coral-like microstructure was synthesized by one-step hydrothermal method, according to XRD, SEM, XPS and UV-Vis DRS results, Co atoms are successfully doped into the ZnS lattice, and the appropriate doping of Co atoms does not change the phase structure and morphology of ZnS. Except for the newly founded hydrothermal synthetic rule in contrast with previous investigations, the Co-doped ZnS samples show a decreased band gap value from 3.32 eV to 2.65 eV with the Co/Zn atomic ratio from 0 to 1, which indicates enhanced absorption of visible light. Furthermore, the doped samples exhibit fluorescence quenching, which is beneficial to photocatalytic performance. The optimal doping level of Co was determined to be 5.49 at.% upon photocatalytic degradation results of the methylene blue under simulated solar light irradiation. Too much or less Co content produces worse photocatalytic result.

As for the morphology control process, two-dimensional (2D)  $\text{LaCO}_3\text{OH}$  microplates and three-dimensional (3D) layer-by-layer (LBL)  $\text{LaCO}_3\text{OH}$  were produced by one-pot hydrothermal method. The different microstructures can be controlled by simply adjusting the addition volume of carbon disulfide ( $\text{CS}_2$ ).  $\text{LaCO}_3\text{OH}$  with different morphologies exhibited different photoluminescence (PL) intensity owing to their different surface defects. In addition, CuS with different morphologies (i.e., hollow sphere and flower-like ones), was synthesized by

hydrothermal method with different addition volume of thiourea in the raw materials, the two CuS morphologies showed distinguished supercapacitor property. Notably, the specific capacitance of CuS hollow sphere is about 3 times of the flower-like CuS. Furthermore, the CuS hollow sphere was moderated with carbon quantum dots in order to obtain larger capacity. As such, the CuS -5% carbon dots was determined to be the optimal composition proportion.

The g-C<sub>3</sub>N<sub>4</sub> modified ZnS composites were prepared through a simple one step calcination process of thiourea and zinc acetate at 600°C in the tube furnace. XRD and XPS results show the successful composition of the two components, indicating that the two components does not interfere with the synthesis of each other. The composites were coated on nickel foam and used as electrodes for supercapacitor tests in 6 M KOH aqueous electrolyte. The composition system exhibited superfine pseudocapacitance performance in this work with the optimal complex of sample ZT0.02, i.e., weight ratio of zinc acetate to thiourea at 0.02.

The composites show good supercapacitor performance in specific capacity, rate capability and cyclic stability. The sample ZT0.02 has specific capacity of 497.1 F g<sup>-1</sup> at 1 A g<sup>-1</sup> current density and 345.9 F g<sup>-1</sup> at 10 A g<sup>-1</sup>, exhibiting high rate capability with the appreciable capacitance retention of 69.5%. The voltage drop of the optimized electrode is 0.12 V, indicating the small impedance of the electrode. The ZT0.02 electrode also has excellent cyclic stability with the capacitance retention of 86% at 2 A g<sup>-1</sup> after 600 charge and discharge cycles and 78% at 5 A g<sup>-1</sup>. The good supercapacitor performance of the system was attributed to the fact that nickel participated in the electrochemical reaction, when the nickel foam was substituted by stainless steel, no redox peaks be detected in this testing condition. Therefore, the g-C<sub>3</sub>N<sub>4</sub>/ZnS electrode should be classified as g-C<sub>3</sub>N<sub>4</sub>/ZnS/Ni system.

**Keywords:** Sulfide; C<sub>3</sub>N<sub>4</sub>; Morphology control; Photocatalysis; Supercapacitor



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