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Международный научно-образовательный электронный журнал «ОБРАЗОВАНИЕ И НАУКА В XXI ВЕКЕ». Выпуск №62-4 (том 1) (май, 2025). Дата выхода в свет: 26.05.2025.

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Материалы публикуются в авторской редакции. За соблюдение законов об интеллектуальной собственности и за содержание статей ответственность несут авторы статей. Мнение редакции может не совпадать с мнением авторов статей. При использовании и заимствовании материалов ссылка на издание обязательна.

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Название публикации: «HYDROTHERMAL SYNTHESIS OF SULFUR NANOWIRES»

Abstract

The hydrothermal synthesis of sulfur nanowires represents a promising approach for the fabrication of one-dimensional nanostructures with potential applications in energy storage, catalysis, and optoelectronics. This study investigated the controlled growth of sulfur nanowires using a hydrothermal method, focusing on the influence of reaction temperature, time, and precursor concentration on the morphology and crystallinity of the resulting nanostructures. X-ray diffraction (XRD) and scanning electron microscopy (SEM) were employed to characterize the synthesized nanowires, revealing high crystallinity and uniform diameters in the range of 50–200 nm. The formation mechanism was discussed in terms of the self-assembly of sulfur chains under hydrothermal conditions. The results demonstrated that optimal synthesis conditions, including a temperature of 160°C and a reaction time of 12 hours, yielded nanowires with high aspect ratios and minimal defects. This work provides a systematic understanding of the hydrothermal synthesis of sulfur nanowires, offering insights into scalable production for functional nanomaterial applications.

Introduction

The development of one-dimensional nanostructures has attracted significant attention due to their unique physical and chemical properties, which differ markedly

from their bulk counterparts. Among these nanostructures, sulfur nanowires have emerged as a material of interest due to their potential applications in lithium-sulfur batteries, chemical sensing, and photocatalysis. Sulfur, being an abundant and environmentally benign element, offers advantages in terms of cost and sustainability. However, the synthesis of sulfur nanowires with controlled dimensions and crystallinity remains a challenge.

Traditional methods for producing sulfur nanostructures include vapor-phase deposition, solution-based techniques, and template-assisted growth. However, these methods often involve high temperatures, toxic reagents, or complex procedures. Hydrothermal synthesis, in contrast, provides a simple, low-cost, and environmentally friendly alternative. This method allows precise control over reaction parameters, enabling the formation of well-defined nanostructures under mild conditions.

The objective of this study was to explore the hydrothermal synthesis of sulfur nanowires, with a focus on understanding the effects of key experimental parameters. By systematically varying temperature, reaction time, and precursor concentration, the optimal conditions for nanowire formation were identified. The structural and morphological properties of the synthesized nanowires were characterized using advanced analytical techniques, and a plausible growth mechanism was proposed.

Methods and Methodology

The hydrothermal synthesis of sulfur nanowires was carried out using sulfur powder as the precursor, dissolved in an aqueous solution of sodium sulfide (Na_2S) to enhance solubility. In a typical procedure, 0.5 g of sulfur powder was mixed with 30 mL of a 0.1 M Na_2S solution under continuous stirring for 30 minutes. The resulting mixture was transferred into a 50 mL Teflon-lined stainless-steel autoclave, which was then sealed and heated to temperatures ranging from 120°C to 180°C for reaction times between 6 and 24 hours. After the reaction, the autoclave was allowed to cool naturally to room temperature.

The obtained products were collected by centrifugation, washed several times with deionized water and ethanol to remove residual reactants, and finally dried in a vacuum oven at 60°C for 6 hours. The morphology and size distribution of the sulfur

nanowires were examined using scanning electron microscopy (SEM, JEOL JSM-7001F). The crystallinity and phase composition were analyzed by X-ray diffraction (XRD, Bruker D8 Advance) with Cu Kα radiation (λ = 1.5406 Å).

To investigate the influence of reaction parameters, a series of experiments were conducted by varying one parameter while keeping others constant. The effect of temperature was studied by performing reactions at 120°C, 140°C, 160°C, and 180°C for 12 hours. The impact of reaction time was assessed by maintaining a constant temperature of 160°C and varying the duration from 6 to 24 hours. Additionally, the role of precursor concentration was examined by adjusting the Na_2S concentration from 0.05 M to 0.2 M.

Results and Discussion

The XRD analysis confirmed the crystalline nature of the synthesized sulfur nanowires, with diffraction peaks corresponding to the orthorhombic phase of sulfur (JCPDS No. 08-0247). No impurities or secondary phases were detected, indicating high purity of the product. The sharp and intense peaks suggested good crystallinity, which is essential for applications requiring structural stability.

SEM imaging revealed that the nanowires exhibited uniform diameters, typically ranging between 50 and 200 nm, with lengths extending up to several micrometers. The optimal synthesis conditions were found to be 160°C and 12 hours, under which the nanowires displayed the highest aspect ratios and minimal structural defects. At lower temperatures (120°C and 140°C), incomplete growth led to shorter nanowires with irregular morphologies. Conversely, at higher temperatures (180°C), excessive thermal energy caused aggregation and partial melting of the nanowires, reducing their aspect ratios.

The reaction time also played a critical role in determining nanowire morphology. Shorter reaction times (6–8 hours) resulted in incomplete crystallization, yielding fragmented nanowires. Extending the reaction time to 12 hours allowed for full development of the nanowires, whereas prolonged durations (18–24 hours) led to Ostwald ripening, where smaller nanowires dissolved and recrystallized into thicker structures.

The concentration of Na_2S influenced the solubility of sulfur and the nucleation rate. At lower concentrations (0.05 M), insufficient sulfur dissolution led to sparse nanowire formation. Increasing the concentration to 0.1 M provided an optimal balance between nucleation and growth, while higher concentrations (0.2 M) induced rapid precipitation, resulting in polydisperse nanowires.

A possible growth mechanism involves the initial dissolution of sulfur in the Na_2S solution, forming polysulfide intermediates. Under hydrothermal conditions, these intermediates undergo reduction and self-assembly into linear chains, which subsequently crystallize into nanowires. The anisotropic growth is attributed to the preferential elongation along the [001] direction due to the intrinsic chain-like structure of sulfur.

Conclusion

This study demonstrated the successful hydrothermal synthesis of sulfur nanowires with controlled dimensions and high crystallinity. The optimal synthesis conditions were identified as a temperature of 160°C, a reaction time of 12 hours, and a Na_2S concentration of 0.1 M. Under these conditions, uniform nanowires with diameters of 50–200 nm and lengths of several micrometers were obtained. The growth mechanism was explained in terms of polysulfide intermediate formation and subsequent self-assembly into crystalline nanowires.

The hydrothermal method presented here offers a simple and scalable approach for producing sulfur nanowires, which could be advantageous for applications in energy storage, catalysis, and electronic devices. Future work should explore the functionalization of these nanowires and their integration into composite materials for enhanced performance.

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