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# A hydrothermal approach to flake-shaped CdS single crystals

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**Abstract**

The morphological transformation process from CdS nanorods to hexagonal CdS flakes was investigated in detail by transmission electron microscope (TEM). The CdS flakes were liable to form with the alkalinity being increased and the reaction time being prolonged. Using this transformation, hexagonal CdS flakes with diameters of 0.3 cm were grown via a recrystallization process in 6 mol/L sodium hydroxide solution at 250 °C. And the formation mechanism of CdS flakes is suggested based on the growth habits of polar crystals under proper basic hydrothermal conditions.


*Keywords:* Crystal morphology; Hydrothermal approach; Crystal growth; Cadmium compounds

## 1. Introduction

The energy band gap of CdS having a hexagonal structure at room temperature has been known to be about 2.42 eV [1] which is in the visible region. CdS crystal, owing to its optical and electrical properties, could be used in photovoltaic cells, radiation detection devices, infrared windows, phosphor and photoconductor research. Therefore, bulk and perfect single crystals of CdS were expected to be rewarding and were widely studied for many years.

CdS crystals have been obtained from many various methods [2–5]. Frerichs et al. prepared it by the reaction between hydrogen sulfide and heated cadmium metal in a tube (>900 °C) [6,7], and mobile gas carried CdS to a cooler portion of the tube where it deposits as single crystal material. Reynolds and Czyzak obtained the crystals through a static resublimation process in which a charge of CdS is heated in an atmosphere of hydrogen sulfide in a sealed tube (>1000 °C) [8–10]. Stanley has used a dynamic method in which a carrier gas is passed over heated CdS powder [11]. Here, using pre-obtained CdS crystalline powders, flake-shaped crystals of CdS with diameters of 0.3 cm were grown via a recrystallization process in 6 mol/L sodium hydroxide solution at 250 °C for 4 days. Compared with the above-mentioned, the reaction conditions in this route are environmental benign and easy to control, and especially, the reaction temperature is lower.

## 2. Experimental section

### 2.1. Synthesis of CdS precursors

All of the chemical reagents used in this experiment were of analytic purity and were purchased from Shanghai Chemical Co. Ltd. without further purification. $CdCl_2$ was stoichiometrically added with $Na_2S$ to a Teflon-lined stainless steel autoclave with a capacity of 35 mL. Then the autoclave was filled with 1 mol/L NaOH aqueous solution up to about 75% of the total volume. The synthesis reaction equation is

$$Cd^{2+} + 2OH^- \rightarrow Cd(OH)_2\downarrow$$

$$S^{2+} + Cd(OH)_2 \rightarrow CdS\downarrow + 2OH^-$$

The autoclave was maintained at 250 °C for 20 h and then allowed to cool to room temperature. A color precipitate was

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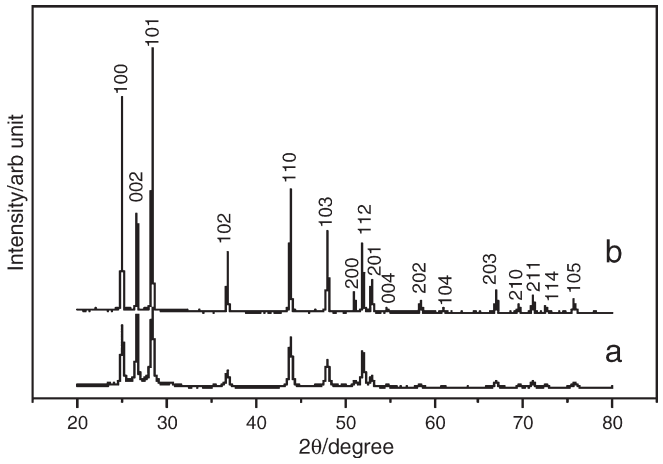


Fig. 1. (a) XRD pattern of the pre-prepared CdS powders. (b) XRD pattern of the flake-shaped crystals of CdS.

collected and washed with absolute ethanol and distilled water to remove residue of $Na^+$ and $OH^-$ ions. The final products were dried at 50 °C for 3 h. The pre-obtained CdS powders were characterized by X-ray powder diffraction (Fig. 1a), and no peaks of impurity phases such as cadmium oxide are observed.

### 2.2. Growth of single cadmium sulfide crystals

In our experiments to synthesize CdS nanorods in sodium hydroxide solution by the reaction of $CdCl_2$ and $Na_2S$, we found hexagonal flakes of CdS crystal were liable to form with the alkalinity being increased and the reaction time being prolonged, as shown in Fig. 2. Therefore, we designed a method to grow flake-shaped single crystals of CdS. An appropriate amount of pre-obtained CdS powders were added to a Teflon-lined autoclave with sodium hydroxide solution (6 mol/L) up to 75% of its capacity (35 mL). The autoclave was heated to 250 °C in 60 min, and maintained at the temperature for 96 h. Then it cooled to room temperature naturally. The hexagonal flakes were collected, and washed several times with distilled water and absolute ethanol to remove impurities. The final products were dried in vacuum at 50 °C for 5 h.

### 2.3. Characterization

The phase and crystallographic structure of the obtained samples were characterized by X-ray powder diffraction (XRD), operating on a Japan Rigaku D/Max-γA X-ray diffractometer equipped with a graphite-monochromatized Cu

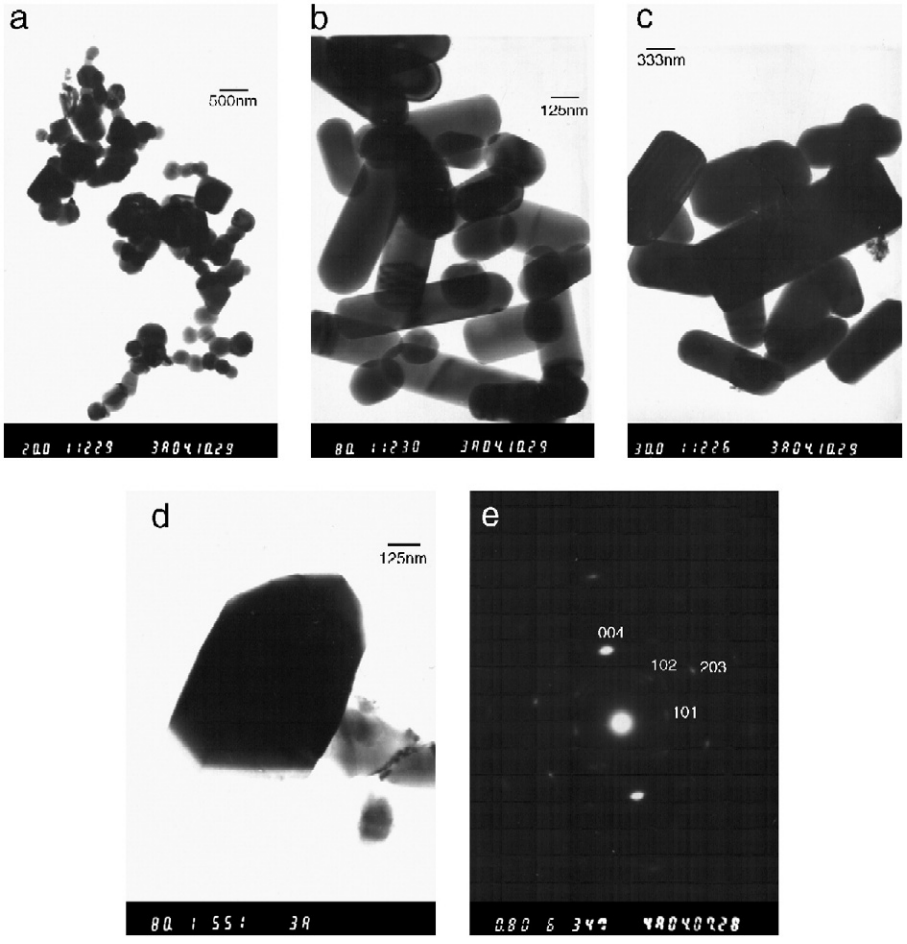


Fig. 2. TEM and ED images of the samples produced by the reaction of $CdCl_2$ and $Na_2S$ in sodium hydroxide solution at 250 °C (a: 1 mol/L NaOH, 20 h; b: 1 mol/L NaOH, 96 h; c: 6 mol/L NaOH, 20 h; d: 6 mol/L NaOH, 96 h), (e) electron diffraction pattern of flake-shaped CdS crystal.