Journal of Solid State Chemistry 262 (2018) 53–57

Contents lists available at ScienceDirect

Journal of Solid State Chemistry

journal homepage: www.elsevier.com/locate/jssc

Synthesis of sub-micro-flakes $CrSe_2$ on glass and (110) Si substrates by solvothermal method

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ARTICLE INFO

Keywords:
Layered structure
Solvothermal method
$CrSe_2(en)_{1/2}$
Substrates
$CrSe_2$ sub-micro-flakes

ABSTRACT

Layered structure MX_2 (M = transition metal, X = S, Se and Te) chalcogenides have rich physic properties and potential applications. While it is still a challenge to prepare the chalcogenides by solvothermal method. In this work, we reported a new solution method to prepare $CrSe_2$ sub-micro-flakes on different substrates. The surface morphologies, structures and compositions of the precursor $CrSe_2(en)_{1/2}$ and $CrSe_2$ were investigated by SEM, XRD, thermogravimetric, IR and Raman spectra. The $CrSe_2$ flakes with the sizes of 5–15 μm were obtained on both glass and (110) Si crystalline substrates. The formation mechanism of $CrSe_2$ sub-micro-flakes is suggested.

1. Introduction

Layered structure MX_2 (M = transition metal, X = S, Se and Te) chalcogenides have variety of optical [1], electric [2], magnetic [3], and catalytic properties [4]. The current on/off ratio of the field effect transistors of the monolayer MoS_2 is as high as 1×10^8 at room temperature [5]. The non-magnetic layered transition-metal temperature [6]. The VS_2 ultrathin nanosheets can be applied in the in-plane supercapacitors [7]. The similarity of the relaxation processes caused by the green or IR lasers Irradiating GeS_2 glass substrate may be a consequence of the principal role played by electron–phonon mechanisms inducing the vanishing of photoinduced phenomena [8].

Among these MX_2 chalcogenides materials, $CrSe_2$ contained 3*d* Cr^{4+} element shows the antiferromagnetic (AF) ground state at room temperature [9,10]. The magnetic property of $CrSe_2$ can be tuned by substituting with V or Ti [11]. The increase of the lattice constants and a gradual passage towards a ferromagnetic state on Ti substitution is observed, and AF order can be maintained at high V replacement [10]. The half-metallic $CrSe_2$ has $Cd(OH)_2$ structure [12] with $a = 3.399$ Å, $c = 5.915$ Å at room temperature. The preparation method of $CrSe_2$ was prepared by oxidation of $KCrSe_2$ at ambient temperature [9]. The compound of $CrSe_2$ is metastable and can be transformed into hexagonal Cr_2Se_3 [13] and Se at around 600 K. The pure $CrSe_2$ cannot be obtained in a stable state due to the unstable Cr^{4+} ion [14].

Recently, some studies have used solvent thermal reaction method to grow two-dimensional materials on different substrates [15–17]. These methods based on the simple and effective way for wet chemistry has been employed to fabricate ZnSe [18], ZnTe [19], and CdS [20]. In this work, we will fabricate $CrSe_2$ via pyrolysizing precursor of $CrSe_2(en)_{1/2}$. The precursor is obtained on the glass and (110) Si substrates by solvent thermal method. The surface morphologies and structures of obtained flakes will be observed. The reaction mechanism of the intermediate products $CrSe_2(en)_{1/2}$ is also discussed.

2. Materials and methods

2.1. Synthesis

2 mmol selenium (Se) and 2 mmol chromium (Cr) powders were added into 30 mL ethylenediamine [21] in 60 mL capacity lined. It was stirred for 10 min with a glass rod. The glass sheet and (110) silicon wafers used as the substrates were respectively cleaned ultrasonically in acetone, ethanol and deionized water for 10 min. After that, two kinds of substrates with the support frame were transferred into the Teflon-lined capacity. Then the mixed solution was put into a Teflon-lined stainless autoclave. The autoclave was sealed and maintained at 200 °C for 2 weeks for synthesis. Then, two kinds of products on the substrates were picked out, cleaned with the ethanol and dried at 70 °C in a vacuum box for 6 h. In order to wipe out the organic matter thoroughly, the precursors were heated up at 300 °C at for 2 h in Ar atmosphere.

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https://doi.org/10.1016/j.jssc.2018.02.020
Received 12 December 2017; Received in revised form 12 February 2018; Accepted 25 February 2018
Available online 27 February 2018
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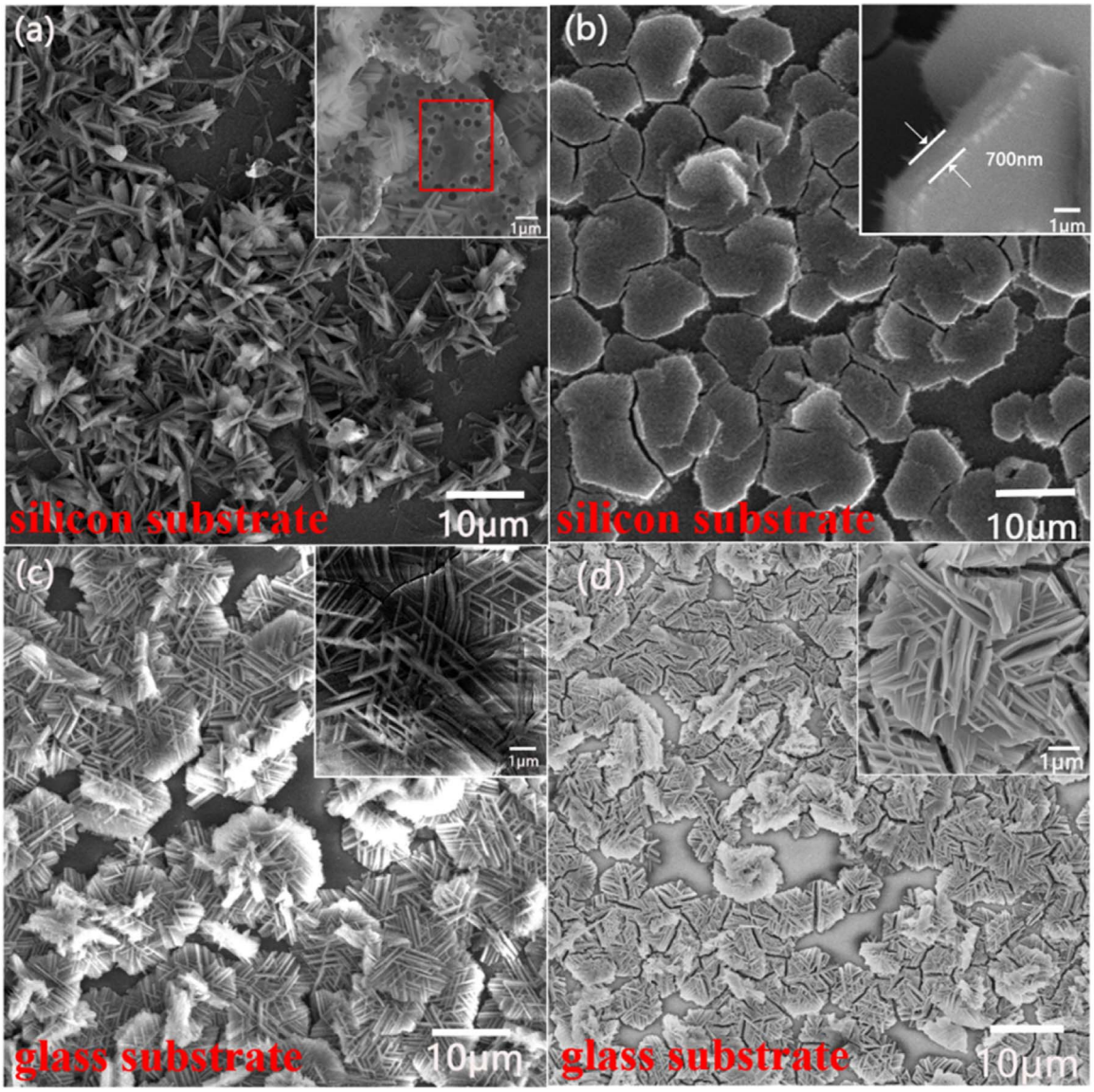

Fig. 1. SEM images of the products on (a) (110) silicon substrate without pyrolysis, (b) (110) silicon substrate after pyrolysis, (c) glass substrate without pyrolysis, (d) glass substrate after pyrolysis. Insets in (a-d) show the partially enlarged images.

2.2. Characterization

Thermogravimetric (TG) curve of the precursor was recorded at a rate of 5 °C/min up to 800 °C under nitrogen protection in Netzsch Synchronous Thermal Analyzer. The X-ray diffraction (XRD) analysis was done with a Rigaku D/max-3C X-ray diffractometer using Cu Kα radiation from 10° to 65°. The images of the products were observed with scanning electron microscopic (SEM) of a FEI-NNS450 and the elemental composition was tested by the EDS. The infrared absorption spectrum was recorded by a Nicolet Nexus Fourier transform infrared spectrometer over a wavenumber range from 400 to 4000 cm^{-1}. Raman spectra (RS) were measured on a Renishaw in Via Raman Microscope (λ *exc* = 532 nm) from 100 to 1000 cm^{-1}.

3. Results and discussion

3.1. The surface morphologies and structures

Fig. 1 shows the microscopic morphologies of the final products on (110) silicon and glass substrates. The SEM image in Fig. 1a shows the product obtained on the (110) silicon substrate without pyrolysis. The sub-micro-flakes attached to many flakes whiskers are covered on the substrate. From the inset in Fig. 1a, the perforated particles indicate that the Cr particles were dissolved at the bottom of the Teflon-lined capacity. The SEM image in Fig. 1b shows the product obtained by heating up the product on (110) silicon substrate without pyrolysis at 300 °C. The burrs on the flakes disappeared and the flakes (5–15 μm in diameter) were perfectly smooth. The thickness of the sheet is about 700 nm in the inset of the Fig. 1b. Fig. 1c shows the image of the product on the glass substrate without pyrolysis. Compared with the product on (110) Si substrate, the flakes and the uniform burrs on the flakes are seen clearly. Fig. 1d shows the image of the produce on glass substrate after pyrolysis obtained by heating up the product on glass substrate without pyrolysis at 300 °C. Similarly, the shape of the sample on glass substrate after pyrolysis are more uniform and seen clearly after pyrolysis to 300 °C. In addition, there are sheet-like structures on the flakes as shown in the inset of Fig. 1d.

In order to determine the compositions of the products after pyrolysis, EDS were measured. Fig. 2a and b show that the mole ratio of Se and Cr is approximately 2:1 on (110) silicon and glass substrates at the pyrolysis temperature 300 °C. It indicates two products contain Se and Cr elements and the corresponding mole ratio is 2:1.

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