Journal of Materials Science and Engineering A 16 (4-6) (2026) 47-52
doi: 10.17265/2161-6213/2026.4-6.001

Controllable Preparation and Optical Anisotropy of High-Quality GeS

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Abstract: Germanium sulfide (GeS) has emerged as a promising candidate for the development of polarization-sensitive photodetectors and nonlinear optical devices, owing to its appealing attributes including optical anisotropy, high carrier mobility, tunable electronic and optical properties, natural abundance, and environmental benignity. Nevertheless, GeS obtained by conventional preparation methods suffers from limited lateral size, irregular morphology, and high defect density. To overcome these drawbacks, herein we report the controllable preparation of high-quality GeS via a spatially confined chemical vapor deposition (SPCVD) approach. The crystal structure and polarized optical properties of the as-grown GeS are systematically investigated. It is revealed that the material possesses a band gap of 1.55 eV and exhibits strong optical absorption in the visible–ultraviolet region. Comprehensive polarization-resolved optical measurements demonstrate an optical polarization ratio up to 1.62. This study offers an effective synthetic strategy for the preparation of high-quality two-dimensional layered GeS crystals, and provides theoretical and experimental support for its applications in polarized optical devices.

Key words: Chemical vapor deposition, optical anisotropy, polarization response, photodetector.

1. Introduction

Two-dimensional germanium-based chalcogenides exhibit great competitiveness in polarization-sensitive devices and energy storage fields compared with transition metal dichalcogenides (TMDs) and black phosphorus, owing to their merits including low-symmetric orthorhombic crystal structure, tunable band gap, environmental friendliness and earth abundance [1-4]. Particularly in the field of polarization detection, their core advantage originates from the remarkable in-plane optoelectronic anisotropy induced by low-symmetry crystal structure, which provides a material basis for integrated polarization detection systems without external polarizers and shows important application prospects in visible to near-infrared polarization remote sensing, medical diagnosis, high-resolution imaging and other fields [5-8]. Among them, germanium sulfide (GeS), as a typical two-dimensional germanium-based chalcogenide, possesses an orthorhombic structure, in which intra-layer atoms are bonded by strong covalent bonds and inter-layers are stacked via weak van der Waals forces. Such structural characteristics endow GeS with significant in-plane anisotropy, laying a foundation for its applications in polarized optical devices [9].

In recent years, researchers have carried out a series of studies on the growth and properties of GeS. For growth methods, various preparation techniques have been developed, including chemical vapor deposition, physical vapor deposition, and mechanical exfoliation [10-13]. For instance, Lan et al. synthesized GeS nanobelts with a thickness of 20-50 nm, a width of several micrometers, and a length of hundreds of micrometers via chemical vapor deposition [14]. At present, chemical vapor deposition has become the mainstream technique for fabricating two-dimensional GeS materials due to its advantages of high crystal quality, good controllability over morphology, and feasibility for large-area growth. However, the chemical vapor deposition preparation of GeS still suffers from

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drawbacks such as a narrow growth parameter window, non-uniform product morphology, and difficulty in precise orientation control. The microscopic mechanism of nucleation and growth of GeS crystals remains unclear, which limits the controllable preparation of high-quality GeS [15, 16]. In the study of polarized optical properties, most existing investigations focus on the characterization of macroscopic optical responses. There is still a lack of systematic and in-depth research on the origin of the polarized optical properties of GeS and the structure–property relationship between crystal structure and polarization characteristics. Therefore, it is urgent to break through this bottleneck through material growth.

Given the above research progress and unresolved issues, this work focuses on the controllable growth mechanism and polarized optical properties of GeS crystals. Spatially confined chemical vapor deposition was utilized to realize the controllable synthesis of GeS crystals. The as-prepared two-dimensional GeS crystals are typical layered semiconductors with a band gap of 1.55 eV. Optical characterization techniques such as polarized microscopy were employed to verify the polarized optical properties of GeS crystals. This work provides novel insights and solutions for advancing the applications of two-dimensional materials in high-performance optoelectronic devices.

2. Experimental Method

2.1 Preparation

The material preparation was mainly carried out using a dual-temperature-zone tube furnace. Before synthesis, commercial 300 nm SiO_2/Si substrates were cleaned with ethanol and deionized water to remove surface impurities. The growth of all GeS samples was performed in the dual-temperature-zone tube furnace via the CVD method under atmospheric pressure. Another quartz boat containing 20 mg of GeS powder (Adamas, 99.999% purity) was placed in the upstream of the second temperature zone of the tube furnace, 8 cm away from the center of the zone. The substrate was positioned downstream at a distance of 17 cm from the GeS source. Argon gas with a flow rate of 100 sccm was used as the carrier gas and introduced into the system. The center of the second temperature zone of the tube furnace was heated to the target temperature (700 °C) within 30 min. After maintaining the target temperature for 5 min to grow GeS samples, the system was naturally cooled down to room temperature in approximately half an hour.

2.2 Characterization

The morphological features of GeS nanosheets were investigated using a bright-field optical microscope (Olympus BX53-P) and a field-emission scanning electron microscope (SEM, Zeiss Sigma500) equipped with an energy-dispersive X-ray spectrometer (EDS). The thickness and surface roughness of the GeS nanosheets were measured by atomic force microscopy (AFM, Bruker Dimension Icon). The crystal structure was characterized using an X-ray diffractometer (XRD, Bruker AXS D8 Advance). Raman spectra were acquired on a confocal Raman microscope (HORIBA LabRAM HR-800) equipped with a 633 nm laser. X-ray photoelectron spectroscopy (XPS, Thermo Scientific ESCALAB 250Xi) was used to analyze the chemical composition and elemental valence states of the GeS nanosheets. High-resolution transmission electron microscopy (HRTEM) was applied to characterize the microstructure, and the measurements were performed on an FEI Talos F200X TEM at an accelerating voltage of 200 kV.

3. Results and Discussion

3.1 Morphology and Structural Characterization

Fig. 1(a) shows the schematic diagram of the dual-temperature-zone tube furnace. The morphology, structure, and properties of the as-synthesized germanium sulfide single crystals were characterized. Fig. 1(b) presents the optical microscopy image of the as-prepared GeS domains with a size of 10-30 μm. The uniform color contrast in the image indicates that the as-synthesized GeS crystals possess excellent crystallization quality. The thickness of the as-prepared domains ranges from 200 to 250 nm, as shown in Fig. 1(c, d). To confirm that the crystals with different morphologies are all GeS, Raman spectroscopy was carried out in this work, and the results are displayed in Fig. 1(e). Four characteristic Raman peaks can be clearly observed for both rectangular and hexagonal crystals. Among them, the peaks at 148.2 cm^{-1} and 212.7 cm^{-1} are assigned to the $B_{3}g$ vibrational modes of the GeS lattice, while the peaks at 236.3 cm^{-1} and 270.8 cm^{-1} correspond to the A_g vibrational modes, which is consistent with previous reports [17]. Furthermore, XPS measurements were performed to investigate the chemical states of surface elements in GeS nanosheets. The presence of Ge and S elements can be readily identified. Fig. 1(f) shows the binding energies of S $2p_{1/2}$ and S $2p_{3/2}$, which are 163.2 eV and 161.8 eV, respectively. The binding energies of Ge $3d_{3/2}$ and Ge $3d_{5/2}$ in the spectrum (Fig. 1(g)) are 32.9 eV and 30.6 eV, respectively. The binding energies in this work show a slight shift compared with previous reports [18]. The possible reason is speculated as follows: impurity molecules (e.g., moisture and carbon contaminants in air) or surface functional groups (e.g., hydroxyl and carboxyl groups) adsorbed on the sample surface alter the electronic bonding states of Ge and S via charge transfer effects, thus leading to the peak shift.

To further reveal the structural characteristics of GeS crystals, X-ray diffraction (XRD) and transmission electron microscopy (TEM) were employed to investigate their internal atomic microstructure. Fig. 2(a) shows a low-magnification TEM image of the as-synthesized GeS crystal. The corresponding selected-area electron diffraction (SAED) pattern exhibits sharp diffraction spots, reflecting the high crystallinity of the two-dimensional domain. Atomic arrangements cannot be clearly distinguished at low magnification, so a local region was selected for high-resolution transmission electron microscopy (HRTEM) imaging, as presented in Fig. 2(b). The measured lattice spacings are 0.283 nm and 0.284 nm, corresponding to the (100) and (010) crystal planes of GeS, respectively. The similar lattice spacings in the two directions contradict the intrinsic anisotropic nature of GeS. Therefore, it is speculated that a small amount of NaCl-type GeS was grown

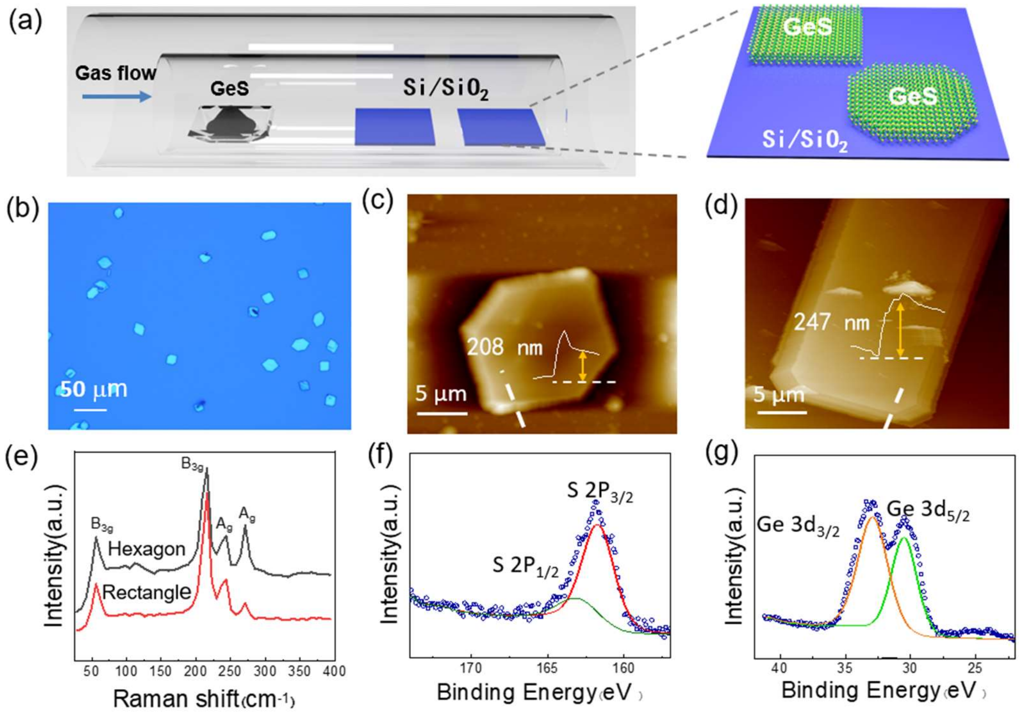

Fig. 1 Qualitative characterization of GeS nanosheets.

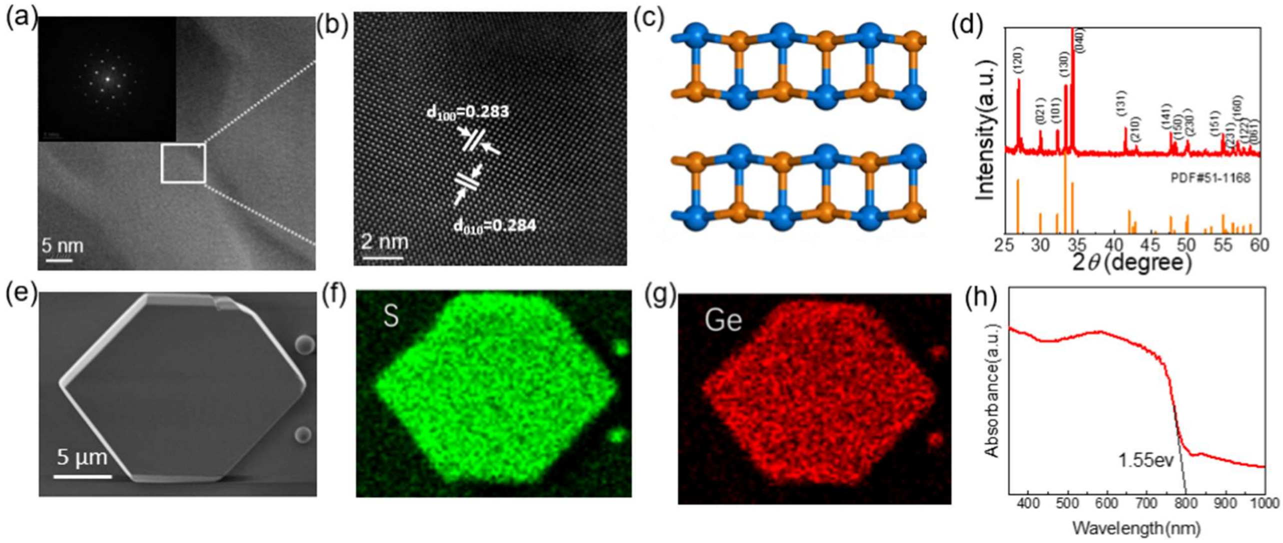

Fig. 2 Structural characterization of GeS nanosheets.

during the experiment, with the atomic structure illustrated in Fig. 2(c). Meanwhile, Fig. 2(d) displays the XRD pattern of the GeS crystal. The experimental result matches well with the standard card of orthorhombic GeS (space group: Pbnm, PDF#51-1168). The three strongest diffraction peaks located at 26.9°, 33.3°, and 34.2° correspond to the (120), (130), and (040) crystal planes of the standard card, respectively. Other diffraction peaks also correspond well to the standard data. The XRD pattern confirms the stable phase structure and high quality of the GeS nanosheets [19]. To further explore the chemical composition and crystal structure of GeS crystals, scanning electron microscopy (SEM) equipped with energy-dispersive X-ray spectroscopy (EDS) mapping was performed. As shown in Fig. 2(e-g), a randomly selected single domain exhibits a perfect hexagonal morphology with a smooth and defect-free surface. The domain is composed of germanium (Ge) and sulfur (S), with uniform elemental distribution. These characterization results demonstrate that the GeS nanosheets grown on SiO_2/Si substrates possess excellent crystallization quality. Fig. 2(h) shows the UV-near-infrared absorption spectrum of the as-prepared GeS nanosheets. The GeS nanosheets exhibit strong optical absorption in the range of approximately 350-800 nm, indicating their potential for broadband photodetection from near-ultraviolet to visible regions. The band gap of GeS nanosheets can be calculated by extrapolating the linear portion to the energy axis, yielding a band gap of 1.55 eV.

3.2 Study on Polarized Optical Properties

According to previous reports, GeS crystals are two-dimensional layered semiconductors with strong in-plane optical anisotropy [20-22]. To further explore its distinctive optical anisotropy, polarized optical properties were investigated using angle-resolved polarized optical microscopy (APROM) and angle-resolved polarized Raman spectroscopy (ARPRS) in this work. The APROM used here was modified from a conventional optical microscope by inserting a polarizer between the light source and the sample to generate polarized light, and an analyzer between the sample and eyepiece to adjust the polarization direction of transmitted light. Thus, APROM can directly visualize the optical anisotropy of anisotropic 2D materials through the optical contrast induced by birefringence. As shown in Fig. 3, the APROM images of a randomly selected GeS crystal exhibit remarkable anisotropy. The polarization angle was adjusted by rotating the substrate, and images were captured every 15°. A clear periodic brightness change of the GeS crystal is observed with varying polarization angles. The sample appears bright at 0°, and the intensity decreases gradually with

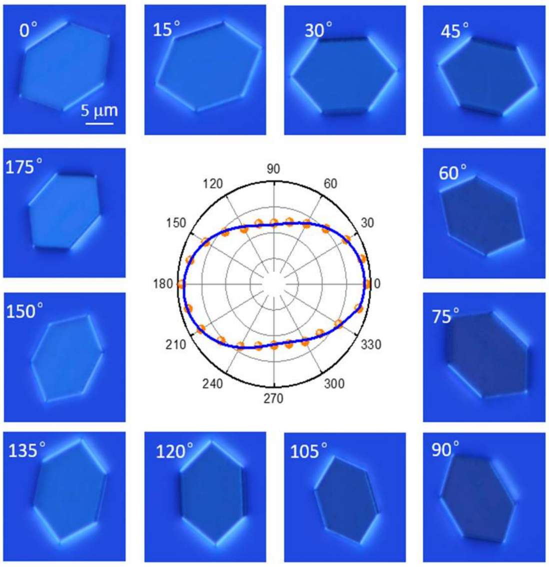

Fig. 3 Polarization-resolved optical microscopy (PROM) characterization of GeS nanosheets.

increasing rotation angle, reaching the minimum at 90°. The intensity recovers only when rotated further to 180°. Meanwhile, the RGB light intensities of the sample were recorded from 0° to 360° and plotted in a polar coordinate diagram. The intensity shows a clear 180° periodic variation: the intensity is 403 nit at 0° and only 249 nit at 90°, giving an anisotropy ratio of 1.62, which is consistent with the visual observation by APROM mentioned above. Based on the above results, it can be concluded that the surface gray scale of GeS crystals exhibits a π-periodic variation and possesses pronounced in-plane anisotropic characteristics.

4. Conclusions

In summary, this chapter developed a spatially confined chemical vapor deposition (CVD) strategy to achieve high-quality synthesis of low-symmetry GeS nanosheets. Characterization results demonstrate that the as-prepared GeS is a typical two-dimensional layered semiconductor with a band gap of 1.55 eV, and exhibits superior properties including high crystallinity, uniform thickness, large domain size, and non-centrosymmetric structure. Anisotropy characterizations reveal that the two-dimensional GeS crystals present distinct polarized optical microscopy (OM) responses with a polarization ratio of 1.62. These findings indicate that two-dimensional GeS crystals hold promising application potential in the development of polarization-sensitive optoelectronic devices.

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