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Large-scale synthesis of van der Waals 1-dimensional material Mo₆S₃I₆ by using a MoI₂ precursor



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1. Introduction

To overcome the problems of existing nanoscale materials, there has been a paradigm shift in research toward low-dimensional (2D and 1D) materials [1-4]. 2D materials have been applied for diverse purposes, ranging from basic materials research to various device applications, because of their advantages, which include atomicunit thickness flexibility, a high surface-to-volume ratio, and simple heterogeneous junctions that are based on van der Waals (vdW) bonding [5,6]. However, dangling bonds are inevitably present at the grain boundaries or edges in the 2D material; this causes problems such as reduced charge mobility [7–9]. Ultimately, it is necessary to develop 1D materials in the form of an atomic-scale linear chain in which there is no such dangling bond (i.e., no edge or grain boundary).

Recently, many studies on various 1D linear materials such as

S. Oh and S. Chae equally contributed to this work.

ABSTRACT

By using a MoI₂ precursor, a one-dimensional linear chain material, $Mo_6S_3I_6$, was synthesized on a centimeter scale. As MoI₂ can be used to form Mo₆S₃I₆ through a gas-phase reaction, a larger amount of single crystals can be synthesized; moreover, these crystals are significantly longer than those synthesized by using Mo, S, and I powder. The synthesized Mo₆S₃I₆ exhibited the characteristics of paramagnetic semiconductors with excellent thermal stability and a low bandgap. Furthermore, $Mo_6S_3I_6$ can be separated into long nanometer-scale chains by a solution dispersion process.

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Mo₆S_{9-x}I_x [10–13], Nb₂Se₉ [14–17], V₂Se₉ [4,18–20], Sb₂Se₃ [21,22], and VS₄ [23–25] have been reported. The 1D linear materials with vdW bonds are obtained by first synthesizing a bulk material with excellent crystallinity, and then obtaining the ultimate atomic linear material through mechanical separation or chemical dispersion [4,14,15,17-20]. Therefore, it is very important to synthesize a large amount of bulk materials with excellent crystallinity. Among these 1D materials, $Mo_6S_{9-x}I_x$, which has a single molecular chain in which sulfur and iodine atoms (Fig. 1a, See the supporting information Table S1 for lattice parameters for Mo₆S₃I₆) are covalently bonded to molybdenum atoms, has excellent thermal stability, is biocompatible, and allows the electrical characteristics from the semiconductor to the metal to be controlled through the adjustment of the ratio of x; thus, various studies ranging from synthesis to application have been conducted [26-28]. Mo₆S_{9-x}I_x is generally synthesized through a stoichiometric solid-state reaction of molybdenum, sulfur, and iodine powder in guartz ampoule at high temperatures [28,29]. However, this synthesis method entails intermediate phases such as $Mo_6S_6I_2$ and MoI_2 ($6Mo + 3S + 3I_2 \rightarrow$ $Mo_6S_6I_2 + MoI_2 + ...$ [30]. The $Mo_6S_6I_2$ phase is involved in a solid state, whereas the MoI₂ phase occurs as a gaseous one and is involved in the synthesis of $Mo_6S_{9-x}I_x$. (In the case of $Mo_6S_3I_6$: $Mo_6S_6I_2(s) + 6Mo(s) + 5I_2(g) \rightarrow 2Mo_6S_3I_6(s), 6MoI_2(g) + 3S(g) \rightarrow$

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Fig. 1. Schematic showing crystal structure (a) and the growth mechanism (b) of Mo₆S₃I₆.

 $Mo_6S_3I_6(s) + 3I_2(g).)$

In the case of MoI₂, which participates in the gas-phase reaction, $Mo_6S_{9-x}I_x$ can be transported away from the source zone and grow without impurities, whereas the $Mo_6S_6I_2$ that participates in the solid-phase reaction remains in the source zone with the synthesized $Mo_6S_{9-x}I_x$ (Fig. 1b). In this study, we first synthesized MoI_2 , and used it as a precursor for the large-scale synthesis of centimeter-scale $Mo_6S_3I_6$ in the transported region. Additionally, it was confirmed that the excellent crystallinity observed on the centimeter scale can be maintained even under the condition of nanoscale dispersion. It is expected that this method can be widely employed for various $Mo_6S_3I_6$ applications.

2. Materials and methods

2.1. Synthesis of MoI₂ and Mo₆S₃I₆

Mol₂ was prepared by using a stoichiometric amount of elements, Mo powder (≥99.9%, 1–5 µm, Aldrich) and iodine (crystalline, 99.99+%, Alfa Aesar). The starting elements were thoroughly mixed, pelletized, and placed in an evacuated quartz tube. Then, the quartz tube was heated to 700 °C for 72 h and allowed to cool to room temperature in a box furnace (See the supporting information Fig. S1 for more details). The resulting product was a dark gray sintered powder. Mo₆S₃I₆ was synthesized by using various molybdenum sources: Mol₂, pure Mo powder (≥99.9%, 1–5 µm, Aldrich), and Mo foil. When Mol₂ was used as a precursor, a mixture of Mol₂ and S (powder, 99.0%, DAEJUNG) with a Mol₂-to-S ratio of 6:3 was sealed in a 14-cm-long evacuated quartz tube. Then, the ampoule was heated for 72 h at a temperature between 1020 and 1120 °C and cooled to room temperature. A large single crystal of $Mo_6S_3I_6$ was transported to the opposite end of the ampoule and mechanically obtained.

2.2. Dispersion

10 mg of $Mo_6S_3I_6$ was dispersed in 10 mL of IPA by using ultrasonication. The ultrasonication process was carried out by operating a probe sonicator (VC 505, Sonics & Materials Inc.) for 5 min at 2-s on/2-s off intervals to roughly break down the bulk crystals and thus improve dispersion efficiency. Then, bath sonication (B2005S-68 K, 68 kHz, 200 W, KODO Technical) was performed for 3 h to exfoliate the crushed bulk $Mo_6S_3I_6$ into nanoscale single chains. After ultrasonication, insufficiently dispersed chains were separated by performing centrifugation at 6000 rpm for 10 min. A total of 5 mL of the supernatant solution was used for AFM analysis.

2.3. Characterization

XRD (D8 Advance, Bruker) was performed by using Cu-K_α radiation ($\lambda = 0.154$ nm). Field-emission scanning electron microscopy (FESEM, Hitachi, S-4300SE) and an attached energy-dispersive spectrometer (EDS) were used for morphological and compositional analysis. Thermogravimetric analysis and differential scanning calorimetry (TGA/DSC, TA Instrument, SDT Q600) measurements were performed at 20 °C·min⁻¹ under the condition of an N₂ atmosphere. A SQUID magnetometer (Quantum Design Inc., MPMS3-Evercool) was used to analyze the magnetic properties under the conditions of a temperature range of 1.8–50 K, and an applied field of 1 T. Atomic force microscopy (AFM, Park systems, NX 10) was performed in non-contact mode on a dispersed Mo₆S₃I₆ wire-coated Si/SiO₂ wafer. A UV–vis

spectrophotometer (Agilent Technologies Inc., Agilent 89090A) was used to measure the UV absorption of the dispersion. A currentvoltage curve was obtained for the temperature range of 100–500 K.

3. Results and discussion

Fig. 2a shows a digital photograph (quartz ampoule) and the XRD patterns of the synthesized materials in the source zone and transport zone when MoI₂ was used as a precursor. In the source zone, the Mo₆S₃I₆ and Mo₆S₆I₂ phases were mixed at a temperature of 1070 °C and above. However, a few MoS₂ phases were observed at a temperature of 1020 °C. On the other hand, in the transport zone, only the pure Mo₆S₃I₆ phase, which corresponded to a thin and long centimeter-scale hair-like shape, was observed. The SEM image of Fig. 2b shows that nanorods, micro-cubes which are act as seed of Mo₆S₃I₆ nanowires (Mo₆S₆I₂), and other micro-sized impurities such as MoS₂ (Fig. 2b and 1020 °C) were mixed in the source zone, whereas a thin and long single phase (Mo₆S₃I₆) was well grown in the transport zone.

Fig. 3a is a photograph of the quartz ampoule that was synthesized by using various precursors. Although it is possible to synthesize Mo₆S₃I₆ by using a pure molybdenum source, such as Mo powder or Mo foil (see the supplementary figure Fig. S2), the shape observed in the transport zone is very different. More specifically, in the case of the MoI₂ precursor, a large amount of centimeter-scale, hair-like Mo₆S₃I₆ was synthesized, whereas when the Mo powder or Mo foil precursor was used, the yield of transported Mo₆S₃I₆ was very low. We measured the transport yield of the Mo₆S₃I₆ synthesized in the transport zone by using the following formula (Fig. 3b). The transport yield was calculated by determining the amount of transported Mo₆S₃I₆ with respect to each initial total mass under the condition that the same mole of Mo was used regardless of the Mo source. In the case of MoI₂, the pure Mo₆S₃I₆ phase occurred at a rate that was 5–15 times higher than that of other metallic Mo sources. The diameter of the Mo₆S₃I₆ synthesized through continuous SEM was also confirmed to be constant (Fig. 3c), and it is expected that very long 1D nano-chains can be obtained when dispersion is performed.

The stability of $Mo_6S_3I_6$ was confirmed by performing thermogravimetric characterization in an N_2 atmosphere (Fig. 4a). The initial mass reduction below 100 °C corresponds to the process by

which adsorbed moisture and residual iodine were removed. The changes occurring near 464 °C were confirmed by the formation of MoO_2 and mass reduction at 762 °C that resulted from the complete decomposition of $Mo_6S_3I_6$ to metallic Mo and Mo_2S_3 phases; this process is illustrated in Fig. 4b as the XRD pattern analysis of heat-treated $Mo_6S_3I_6$ samples at 500 °C and 800 °C. Thus, $Mo_6S_3I_6$ was confirmed to be a very stable material up to 450 °C (The results of other experiments also show that $Mo_6S_3I_6$ is stable in the ambient atmosphere [28]).

The electrical properties of the synthesized Mo₆S₃I₆ (100-500 K) were observed to be dependent on the temperature (Fig. 4c). As the temperature increased, the electrical conductivity increased. The electrical conductivity is known to be proportional to the mobility and concentration of the charge. Considering that mobility was assumed to be constant, the results of Arrhenius fitting (slope: $-E_g/k_BT$, where E_g is the bandgap, k_B is the Boltzmann constant, and *T* is the absolute temperature) were most similar to the measured results corresponding to the 0.159 eV bandgap (Fig. 4d) [29]. More specifically, the $Mo_6S_3I_6$ is a material with a very small bandgap, and it can be inferred that the transition from the valence band to the conduction band is active even at room temperature. The reason for the discrepancy between the measured value and fitting value in the low-temperature region is that the concentration of the charge is low when the temperature is low; therefore, there are few collisions between the charges, and the absolute mobility is large.

The results of high-strength magnetization (1 T) of the synthesized Mo₆S₃I₆ demonstrate an upturn below 50 K that infers the presence of paramagnetic centers (Fig. 5a) [31]. The central atom of $Mo_6S_3I_6$, Mo, is 2+ (4d⁴), and is surrounded by octahedrally symmetric S or I atoms. Considering the arrangement of the four electrons in the Mo 4d orbital, four unpaired electrons are generated when the value of 10 Dq (i.e., the gap between e_g and t_{2g}) is very small according to the crystal field theory. Therefore, the synthesized Mo₆S₃I₆ is applicable to the case in which the value of 10 Dg is large, and it can be deduced that a paramagnetic characteristic exists in which there are one pair of paired electrons and two unpaired electrons. Raman analysis of the synthesized Mo₆S₃I₆ was performed to analyze the crystallinity (Fig. 5b). It can be seen that the measurement results are consistent with the previously reported results for Mo₆S₃I₆ peaks [32]. As shown in Figs. 4 and 5, the Mo₆S₃I₆ that was synthesized by using MoI₂ as a precursor had



Fig. 2. (a) Optical image of quartz ampoule after reaction, and XRD patterns of samples in each zone; source zone (left) and transport zone (right). (b) SEM images of source zone (left) and transport zone (right) for *T* ranging from 1020 to 1120 °C. (MoI₂-to-S ratio: 6:3).



Fig. 3. (a) Optical image of quartz ampoules and transported Mo₆S₃I₆ with different Mo sources. (b) Transport yields for different Mo precursors. (c) Millimeter-scale SEM image of Mo₆S₃I₆ synthesized by using MoI₂.



Fig. 4. (a) TGA/DSC analysis under the conditions of an N₂ atmosphere and a rate of 20 °C h⁻¹. (b) XRD patterns of Mo₆S₃I₆ after heat treatment at 500 and 800 °C. (c) Linear I-V curve for exfoliated Mo₆S₃I₆ wire, with *T* ranging from 100 to 500 K (inset is an SEM image of Mo₆S₃I₆ nanowire and an electrode). (d) Electrical conductivity of Mo₆S₃I₆.



Fig. 5. (a) Results of high-strength magnetization (1 T) of Mo₆S₃I₆ for temperatures ranging between 1.8 and 50 K. (b) Raman spectrum of Mo₆S₃I₆,



Fig. 6. Dispersion and mechanical exfoliation. (a) AFM image of an Mo₆S₃I₆ chain deposited on the Si/SiO₂ substrate, and the height profiles corresponding to each dashed line. (b) UV absorption of dispersed Mo₆S₃I₆ in IPA (inset shows the Tyndall effect occurring in the Mo₆S₃I₆ dispersed solution) and (c) TEM images of dispersed Mo₆S₃I₆ (inset: fast Fourier transform pattern).

excellent and reliable material properties.

The synthesized bulk $Mo_6S_3I_6$ was mechanically exfoliated and the size of the nano-chains was analyzed by using AFM, as shown in Fig. 6a. It can be confirmed that the 1D-structured $Mo_6S_3I_6$ chains with an average diameter of 5–8 nm were well exfoliated and were microscale in length. Also, the bulk $Mo_6S_3I_6$ was chemically dispersed in isopropyl alcohol, sonicated, and then filtered. The solubility and nano-dispersion of $Mo_6S_3I_6$ chains was analyzed by using UV–vis–NIR spectroscopy (Fig. 6b). The strong UV–vis absorption peak and Tyndall effect (inset of Fig. 6b) indicate that nano-dispersion of $Mo_6S_3I_6$ occurred. The dispersed $Mo_6S_3I_6$ nanochain was analyzed by TEM (Fig. 6c) which shows van der Waals bonding characteristics and interatomic chain (0.958 nm). It is suggested that 1D $Mo_6S_3I_6$ can also be dispersed as mono-chains by selecting a dispersant and optimizing the dispersion process.

4. Conclusions

In this study, by using MoI₂ as a precursor to synthesize a 1D linear atomic-chain material, we proposed and experimentally proved that centimeter-scale $Mo_6S_3I_6$ can be synthesized. When MoI₂ is used, it is transported through the gas-phase reaction to begin the $Mo_6S_3I_6$ formation reaction; thus, very long single-crystal $Mo_6S_3I_6$ can be synthesized in a large amount. In fact, a yield that is at least five times more than that obtained by using Mo, S, and I powder to initiate the solid-phase-reaction-based synthesis was confirmed. The synthesized $Mo_6S_3I_6$ can be separated into long nanoscale chains via a solution dispersion process. Therefore, this study demonstrates that various applications of 1D-structured $Mo_6S_3I_6$ are possible.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jallcom.2019.06.287.

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