Chemical Vapor Deposition of IrTe₂ Thin Films

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Abstract: Two-dimensional (2D) IrTe₂ has a profound charge ordering and superconducting state, which is related to its thickness and doping. Here, we report the chemical vapor deposition (CVD) of IrTe₂ films using different Ir precursors on different substrates. The Ir(acac)₃ precursor and hexagonal boron nitride (h-BN) substrate is found to yield a higher quality of polycrystalline IrTe₂ films. Temperature-dependent Raman spectroscopic characterization has shown the q_{1/8} phase to HT phase at ~250 K in the as-grown IrTe₂ films on h-BN. Electrical measurement has shown the HT phase to q_{1/5} phase at around 220 K.

Keywords: chemical vapor deposition (CVD); two-dimensional material; IrTe₂; iridium acetylacetonate; phase transition

1. Introduction

IrTe₂ has attracted much interest for its intriguing properties such as structure phase transitions, charge density wave (CDW) ordering and superconductivity arising from the strong spin-orbit coupling (SOC) [1–6]. With these unique properties, IrTe₂ can be used in memory, oscillator, superconductor devices [7–9], etc.

IrTe₂ has a layered hexagonal structure and each layer is a sandwich-like structure with three layers of Te-Ir-Te atoms. At room temperature, IrTe₂ has a trigonal phase (high temperature phase, HT), in which each iridium atom is coordinated with six tellurium atoms, forming edge-shared IrTe₆ octahedrons [10]. As the temperature lowers to about 280 K, the crystal structure changes from the trigonal phase to the monoclinic phase. In the monoclinic phase, charge ordering is formed due to the charge transfer from Ir 5d to Te 5p, forming Ir⁴⁺-Ir⁴⁺ dimers. This dimerization suppresses the structure of the IrTe₆ octahedron. This monoclinic phase is also called the q_{1/5} phase, in which Ir³⁺ gives an electron to Te₂³⁻, forming the periodic structure of the 33344 (3 refers to Ir³⁺, 4 refers to Ir⁴⁺) arrangement of Ir atoms (Figure 1b) with the modulation vector $\vec{q} = (1/5, 0, -1/5)$ (Figure 1e). As temperature further lowers down to 180 K, the periodic structure of 34433344 forms, i.e., the q_{1/8} phase with the modulation vector $\vec{q} = (1/8, 0, -1/8)$ appears (Figure 1c,f) [11]. In the warming process, IrTe₂ changes from the q_{1/8} phase to the HT phase directly without an intermediate q_{1/5} phase, which is due to the extra pinning energy that existed in the q_{1/8} phase. Phase transition is accompanied by a change of resistance and magnetic susceptibility, which is of great research interest [12].

Additionally, IrTe₂ has a metastable superconducting state. In IrTe₂, the striped charge order competes with the superconducting state [13]. The phase transition from the HT phase to the striped charge-ordered phase of IrTe₂ would be suppressed as the sample thickness decreases, which is favorable for the appearance of the hexagonal superconductive phase [14]. Besides, Pt or Pd doping can also suppress the charge order phase, making the superconductive phase appear [15]. IrTe₂ is

diamagnetic, which is anormal for metallic compounds. This is because that the valence state of Ir^{3+} can form more closed shells. When $T < T_c$ (CDW transition temperature, ~ 280 K), diamagnetism is further strengthened because the density of state near E_F decreases, reducing the Pauli paramagnetism [16,17].

Figure 1. Crystal structure of IrTe_2 in (a,d) high temperature (HT) phase, (b,e) $1/5$ phase and (c,f) $1/8$ phase. Only Ir atoms are shown in the structures. (a–c) are views perpendicular to the ab plane; (d–f) are side views. Ir^{4+} – Ir^{4+} dimers are connected with yellow lines, other Ir–Ir bonding is shown in black lines.

Growth of IrTe_2 single crystals is reported using the chemical vapor transport (CVT) [10] and self-flux technique [13,16,18]. Mechanical exfoliation is further used to obtain nanometer thick IrTe_2 [14]. Mechanical exfoliation is of low yield and poor reproducibility. CVD can grow large-area 2D materials with high uniformity. Graphene and transition metal chalcogenides (TMDs) have been grown by CVD [19–24], showing excellent physical properties similar to those obtained from mechanical exfoliation.

Here in this work, we have successfully synthesized the IrTe_2 thin films using the CVD method. Different Ir precursors (including $\text{Ir}(\text{acac})_3$, IrCl_3 and Ir) and different substrates (including h-BN, SiO_2/Si , mica and sapphire) have been investigated. At optimized conditions, polycrystalline IrTe_2 films with thickness of ~ 20 nm and domain size of ~ 200 nm have been obtained on h-BN. Temperature-dependent Raman spectroscopic characterization demonstrated the transition from the $q_{1/8}$ phase to the HT phase at ~ 250 K. Electrical measurement has also shown the phase transition from the HT phase to the $q_{1/5}$ phase at around 220 K.

2. Materials and Methods

2.1. Materials

IrCl_3 (99.99%) and Te (99.999%) were purchased from Beijing Inokai Technology Co., Ltd., Beijing, China. $\text{Ir}(\text{acac})_3$ ($\geq 97\%$) was purchased from Shanghai Aladdin Biochemical Technology Co., Ltd., Shanghai, China. Ir (99.95%) was purchased from Beijing Zhongnuo New Material Technology Co., Ltd., Beijing, China. All the starting materials were analytical grade and were used without further purification.

2.2. CVD Growth of IrTe_2 Films

Hexagonal-BN (h-BN) crystalline powders (PT110, Momentive) were mechanically exfoliated on SiO_2/Si wafers to prepare h-BN flakes. The h-BN flakes on SiO_2/Si were annealed at 500°C for 1–2 h in air to remove the possible polymer residues. Sapphire flakes were annealed at 1000°C for 1–2 h. Mica flakes were freshly peeled before usage.

2.2.1. IrTe₂ Growth Using Ir(acac)₃ Precursor

An alumina boat containing Ir(acac)₃ precursors (10–20 mg) was put outside of the furnace and heated to 170 °C. Another alumina boat containing Te (~20 g) powder was placed in the upstream of the furnace at a temperature of ~500 °C. Substrates were placed in the center of the furnace with a temperature of 700 °C. 20 SCCM Ar and 20 SCCM H₂ was used as the carrier gas and the total pressure was maintained at ~5 kPa. The experimental conditions including reaction time, reaction temperature, Ir precursor heating temperature, flux rate and proportion of carrier gas were optimized for growth.

2.2.2. IrTe₂ Growth Using IrCl₃ Precursor

An alumina boat containing Te (about 4 g) was put in the first temperature zone of the two-temperature-zone furnace at 500 °C. Another alumina boat containing SiO₂/Si substrates with IrCl₃ was placed in the second temperature zone at a temperature of 700 °C (Figure S3a). 100 SCCM Ar and 20 SCCM H₂ was used as the carrier gas and the total pressure was maintained at atmospheric pressure.

2.2.3. IrTe₂ Growth Using Element Ir as the Precursor

Metal iridium films of about 20 nm thickness on SiO₂/Si wafers were deposited by the vacuum-evaporating method. An alumina boat containing Te (about 4 g) was put in the first temperature zone of the two-temperature-zone furnace at 500 °C. Another alumina boat containing substrates was placed in the second temperature zone at a temperature of 800 °C (Figure S3b). 100 SCCM Ar and 20 SCCM H₂ was used as the carrier gas and the total pressure was maintained at atmospheric pressure.

2.3. Characterization

Scanning Electron Microscopic (SEM) images were taken on a Hitachi S4800. AFM images were captured on a veeco M-Pico microscope. Raman spectra were measured on a home-built vacuum, variable temperature, low-wavenumber Raman system with 532 nm excitation. An attoDry 800 optical stat (attocube systems AG, Germany) was used for sample cooling. A NA = 0.82 low temperature objective (LT-APO/VIS/0.82, attocube systems AG, Germany) was used for laser focusing and signal collection. In the electrical measurement, IrTe₂/h-BN flakes were transferred on a new SiO₂/Si substrate using a PDMS stamp and then a copper TEM grid was used as a shadow mask for the metal contact evaporation. A total of 5 nm Ti and 50 nm Au was electron-beam evaporated for contacts. Low temperature resistance measurements were performed in a physical property measurement system (PPMS, Quantum Design, Inc., San Diego, CA, USA) under He-purged conditions.

3. Results and Discussion

The CVD setup for IrTe₂ growth using Ir(acac)₃ as precursor is shown in Figure 2a. Figure 2b–e show the SEM images of as-grown IrTe₂ films on h-BN, SiO₂/Si, sapphire and mica substrates. The domain size of IrTe₂ films grown in h-BN substrate is about ~200 nm, which is larger than that on other substrates. This is because the atomic smoothness and free dangling of bonds of the h-BN surface minimized the nucleation sites and promoted in-plane epitaxy of IrTe₂ flakes [21,25,26]. However, SiO₂/Si, sapphire and mica lack this advantage, and only smaller crystal domains were obtained on these substrates. Moreover, the as-grown IrTe₂ films using Ir(acac)₃ as precursor showed a larger grain size and a thinner thickness (~20 nm, inset of Figure 2b) than using inorganic precursors (IrCl₃, Ir) (Figure 3). The E_g mode at ~128 cm⁻¹ and A_{1g} mode at ~165 cm⁻¹ are observed from all as-grown IrTe₂ films (Figure 3c).

Figure 2. (a) Illustration of tube furnace for the growth of IrTe₂ films; (b–e) SEM images of as-grown IrTe₂ films on h-BN, SiO₂/Si, sapphire and mica substrates, (f) XRD and (g) EDS spectra of as-grown IrTe₂ films on h-BN adopting Ir(acac)₃ precursor.

Figure 3. (a,b) SEM images of as-grown IrTe₂ films using (a) IrCl₃ and (b) Ir precursors, (c) Raman Spectra of as-grown IrTe₂ using Ir(acac)₃ (red line), IrCl₃ (black line) and Ir (blue line) precursors.

The X-Ray Diffraction (XRD) peaks of as-grown IrTe₂ film on h-BN are shown in Figure 2f. The diffraction peaks at 2θ of 16.4°, 26.8°, 31.0°, 33.18°, 48.0°, 50.8° can be assigned to (001), (100), (101), (002), (110), (111) crystal planes (Figure S4), respectively, which is consistent with the trigonal structure of IrTe₂ [27]. The Energy Dispersive Spectrum (EDS) measurement revealed a Te/Ir ratio of 2.08 in the as-grown IrTe₂ film (Figure 2g), close to the ideal ratio of 2.

Temperature-dependent Raman spectroscopic characterization was carried out to study the phase transitions in the as-grown IrTe₂ films on h-BN. At room temperature, IrTe₂ has a trigonal symmetry, and there are two characteristic Raman peaks: E_g (~128 cm⁻¹) and A_{1g} (~165 cm⁻¹) [28]. As shown in Figure S5, E_g and A_{1g} modes refer to the intralayer and interlayer vibration of Te atoms, respectively.

Figure 4b shows the 2D Raman image of as-grown IrTe₂ film on h-BN in the temperature range of 4–300 K. The fitted peak position and intensity of E_g and A_{1g} modes are shown in Figure 4c,d. At low temperatures, the crystal structure of IrTe₂ is monoclinic, and E_g splits to two peaks: E_g¹ (~124 cm^{−1}), E_g² (~132 cm^{−1}) and A_{1g} splits to A_{1g}¹ (~161 cm^{−1}), A_{1g}² (~172 cm^{−1}) (Figure 4a) due to the symmetry breaking. As the temperature increased up to 250 K, both E_g and A_{1g} modes showed a single Lorentzian peak (Figure 4a). Moreover, the splitting of E_g and A_{1g} modes can only be observed when T < ~250 K, so the phase transition temperature should be at about 250 K (Figure S6), which is consistent to that in previous literature [29]. In addition, the Raman peak positions of E_g and A_{1g} shifted to lower frequency at ~123 cm^{−1} and ~161 cm^{−1}, respectively. In addition, the intensity of A_{1g} and E_g modes increased significantly (Figure 4d). This is because of the strengthening of Te-Te and hence increase of electronic polarizability at high temperatures [28]. We did not observe the Raman feature change in the warming process at about 180 K for the phase transition from q_{1/8} to q_{1/5} phase. This phase transition is also not expected in the warming process because of the high pinning energy of the q_{1/8} phase [10].

Figure 4. (a) Raman spectra of as-grown IrTe₂ film on h-BN using Ir(acac)₃ precursor at 250 K (magenta line) and 180 K (black line). The red, green, blue and cyan solid lines are the fitted peaks using Lorentz functions. (b) Variable-temperature Raman spectra of as-grown IrTe₂ film (warming process). (c) Raman shift and (d) intensity of E_g and A_{1g} modes as a function of temperature.

The q_{1/8} to HT phase-transition temperature in the as-grown IrTe₂ film (~250 K) is lower than that in the literature (280 K) [28]. This may be due to the fact that the crystal domain in our samples is smaller than that in the literature, which affects the phase transition from the q_{1/8} phase to the HT phase. We also collected the temperature-dependent Raman Spectra of as-grown IrTe₂ film on SiO₂/Si, and no Raman change was observed as temperature increased from 4 K to 300 K, which may be due to the much lower quality of the IrTe₂ thin film grown on SiO₂/Si (Figure S7).

A two-terminal device was fabricated to measure the electric property of as-grown IrTe₂ films on h-BN (Figure 5a). As shown in Figure 5b, the electrical measurement result shows that the resistance of the as-grown IrTe₂ thin film decreased in the cooling process from 300 K to 225 K, and was stable at 225 K. Then as the temperature lowers further, the R increased from 210 K to 150 K. When the

temperature reached 150 K, the resistance reached a peak value, and then decreased for $T < 150$ K. There was no obvious phase transition from temperature resistance measurement for the as-grown IrTe₂ thin film on SiO₂/Si (Figure S8), which may be due to the much lower quality of IrTe₂ crystal grown on SiO₂/Si.

Figure 5. (a) Illustration of a two-terminal IrTe₂ device used in temperature-dependent resistance (R-T) measurement (cooling process). (b) R-T and (c) dR/dT-T curves of as-grown IrTe₂ film on h-BN using Ir(acac)₃ precursor.

The resistance increase from 210 K to 150 K is correlated to the HT phase to $q_{1/5}$ phase transition [12]. In the $q_{1/5}$ phase, Ir⁴⁺ forms the local spin-orbit Mott state, increasing the gap of the Hubbard gap. The DOS (Density of States) at E_F of the $q_{1/5}$ phase is lower than that of the HT phase due to the break of the Te₂^{3−} polymeric bond [30]. Then, the resistance of the $q_{1/5}$ phase is higher than that of the HT phase. The $q_{1/5}$ to $q_{1/8}$ phase transition expected at around 180 K was not observed. This may be due to defects and impurities in polycrystalline IrTe₂ film, which can inhibit the phase transition.

4. Conclusions

In this paper, we report CVD growth of IrTe₂ thin films. At optimized conditions, poly crystalline IrTe₂ films with a thickness of ~20 nm and domain size of ~200 nm have been obtained on h-BN using Ir(acac)₃ as the precursor. Temperature-dependent Raman spectroscopic characterization has shown the $q_{1/8}$ phase to HT phase at ~250 K (warming process), and electrical measurement has shown the HT phase to $q_{1/5}$ phase at around 220 K (cooling process). The phase transition temperature of as-grown IrTe₂ thin films is slightly lower than that in the literature for the bulk materials (280 K) [28].

Supplementary Materials: The following are available online at <http://www.mdpi.com/2073-4352/10/7/575/s1>, Figure S1: Vapor pressure of iridium (III) acetylacetonate in 140–210 °C, Figure S2: Vapor pressure of Te in 360–540 °C, Figure S3: Illustration of growth setups for the growth of IrTe₂ films using IrCl₃ and Ir precursors. Figure S4: Crystal plane of IrTe₂ corresponding to diffraction peaks. Figure S5: E_g and A_{1g} vibration modes of IrTe₂. Figure S6: Raman spectra of as-grown IrTe₂ film on h-BN using Ir(acac)₃ precursor from 170 K to 260 K (warming process). Figure S7: Variable-temperature Raman spectra of as-grown IrTe₂ film on SiO₂/Si using Ir precursor. Figure S8: R-T curves of as-grown IrTe₂ films on SiO₂/Si using Ir as precursor.

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Conflicts of Interest: All of the authors declare no conflict of interest.

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