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1D p-type molecular-based coordination polymer semiconductor with ultrahigh mobility

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ABSTRACT

One-dimensional (1D) semiconductor nanostructures exhibit exceptional performance in mitigating short-channel effects and ensuring low power consumption. However, the scarcity of high-mobility p-type 1D materials impedes further advancement. Molecular-based materials offer high designability in structure and properties, making them a promising candidate for 1D p-type semiconductor materials. A molecular-based 1D p-type material was developed under the guidance of coordination chemistry. Cu-HT (HT is the abbreviation of p-hydroxy thiophenol) combines the merits of highly orbital overlap between Cu and S, fully covered surface modification with phenol functional groups, and unique cuprophilic (Cu–Cu) interactions. As such, Cu-HT has a remarkable hole mobility of $27.2 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$, which is one of the highest reported values for 1D molecular-based materials to date and even surpass those of commonly used amorphous silicon as well as the majority of 1D inorganic materials. This achievement underscores the significant potential of coordination polymers in optimizing carrier transport and represents a major advancement in the synthesis of high-performance, 1D p-type semiconductor materials.

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1. Introduction

In microelectronics, the allure of one-dimensional (1D) nanostructured semiconductor materials is undeniable, owing to their exceptional physical and chemical properties [1–4]. In particular, as the channel materials in field-effect transistors (FETs), they exhibit a spectrum of advantages, such as effectively suppressed short-channel effects, low power consumption, and minimized carrier transition effect from the transverse channel electric field. Both n-type and p-type semiconductor materials with high mobility are indispensable in the application of p–n junctions [5,6] and logic circuits [7,8]. Nonetheless, the current state of p-type 1D semiconductors, particularly those exhibiting high mobility, presents a notable challenge in the ongoing development and advancement of 1D semiconductor device technology [9,10].

Molecular-based materials, such as organic polymers, small molecules, organic–inorganic hybrids, and coordination polymers, present a compelling prospect for 1D p-type semiconductor mate-

rials because of their exceptional versatility in structure and property engineering [11–15]. However, these materials commonly suffer from low hole mobility, which is primarily due to the lack of efficient carrier transport pathways and high hole injection barriers [16,17]. Coordination polymers offer a unique avenue for enhancing these properties by incorporating metallic elements to create structures with hybridized molecular orbitals. The judicious selection of metal ions and organic ligands within coordination polymers can lead to increased orbital overlap, facilitating the establishment of effective carrier transport channels and, consequently, enhancing carrier transport efficiency [18–20]. This can be informed by the Hard and Soft Acid and Base (HSAB) theory, which suggests that soft acids and soft bases are prone to extensive orbital overlap [21–23]. Additionally, the modification of functional groups can act as a regulatory mechanism for carrier transport barriers. For example, functional groups with strong electron-donating ability can elevate the highest occupied molecular orbital (HOMO) energy level, thereby effectively reducing the hole injection barrier [24–27]. Furthermore, the distinctive metal-to-metal interaction present in coordination polymers can promote the carrier transport ability [28,29]. The aforementioned factors can lead

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to the formation of extended carrier transport channels, which can improve the overall conductivity and mobility.

In this study, a novel p-type semiconductor material, Cu-HT (HT is the abbreviation of p-hydroxy thiophenol), was synthesized with a nanorod morphology considering the strategic selection of copper ions (classified as soft acid) and p-hydroxy thiophenol ligand (recognized as soft base). The structure of Cu-HT was been meticulously crafted to optimize carrier mobility: (1) Cu ions and S form were configured to create a 1D inorganic conductive chain, leveraging their soft acid and soft base properties to achieve optimal orbital overlap, which is critical for effective charge transport; (2) the surface of the conductive chains was functionalized with phenol functional groups, which are endowed with strong electron-donating properties; and (3) interestingly, a rarely reported cuprophilic (Cu–Cu) interaction was observed in Cu-HT. In the FET test, the hole mobility of Cu-HT nanorods reached as high as $27.2 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$, which is one of the highest values reported to date for 1D molecule-based materials and even surpasses the mobility of commonly used amorphous silicon as well as the majority of 1D inorganic materials [30–35].

2. Experimental

A facile method was developed for the synthesis of Cu-HT. Fig. S1 (online) shows the schematic diagram of the synthesis of Cu-HT nanorods. About 10 mg of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ was dissolved into 40 mL of ethanol, gradually added with 40 μL of HT ligand, and magnetically stirred at 30°C . Large amount of Cu-HT nanorods with 50–100 nm diameters were obtained in 15 min (Fig. 1a). This approach is notably more efficient than the conventional hydrothermal synthesis of coordination polymers, which typically necessitates stringent reaction conditions and prolonged reaction periods [36,37]. The rapid nucleation rate of copper (Cu) and sulfur (S) in the synthesis of Cu-HT nanorods poses a significant challenge in achieving single-crystal with large dimensions. To address this challenge and facilitate the production of Cu-HT nanorods with enhanced dimensions, an in-depth investigation was conducted to elucidate the effects of temperature, stirring condition, and modifiers on the size of the synthesized nanorods (Table 1 and Fig. S2 online).

In the synthesis of Cu-HT nanorods, low temperatures favor the formation of nanorods with larger diameters. However, when stirring was omitted from the synthesis process, Cu-HT nanorods exhibited a large diameter but with less uniform size distribution. The presence of amines in the reaction can compete with the ligands, thereby affecting the dimensional properties of the synthesized product [38]. Triethylenediamine has been identified as an effective regulator in this process. Extensive parallel controlled experiments have demonstrated that both excessive and insufficient amounts of triethylenediamine are detrimental for synthesizing Cu-HT nanorods. The optimal concentration of triethylenediamine for achieving the desired product characteristics with larger size is approximately 50 mmol L^{-1} . Experimental details and characterization data as well as the crystallographic data of Cu-HT (CCDC: 2334489) are shown in Supplementary materials (online).

3. Results and discussion

As evidenced by the energy dispersive spectroscopy (EDS) elements mapping analysis, the elements have a uniform distribution on the nanorods, with an atomic ratio of Cu to S extremely close to 1:1 (Fig. S3 online). The crystallographic structure of Cu-HT was elucidated using powder simulation, with detailed methodology provided in the Supplementary materials (online). The

crystallographic and powder refinement data for Cu-HT are presented in Table S1 (online) with the Rietveld refinement profile for the final cycle in Fig. S4 (online). The simulated crystal reveals that each Cu is bonded to three S, forming a distorted spatial triangular CuS_3 configuration. The Cu–S bond distance is $2.30(3) \text{ \AA}$, with Cu–S–Cu angles ranging from 102.37° to 129.67° . These CuS_3 triangles are interconnected through shared vertices, creating an infinite Cu_3S_3 chain that extends along the *c*-axis crystallographic direction (Fig. 1b). The phenol functional group with strong electron donating ability is fully covered to Cu_3S_3 chains through C–S covalent bonding to form a Cu-HT chain. Such Cu-HT chain is further stacked into a molecular-based material Cu-HT through van der Waals interactions (Fig. 1c). According to a previous research, cuprophilic interactions are observed when the interatomic distance between Cu atoms ranges from 2.4 to $3.0 \text{ \AA}$ [39,40]. In the case of Cu-HT, the interatomic distance between adjacent Cu atoms is measured at $2.67 \text{ \AA}$, which falls within the typical range for cuprophilic interactions. These interactions are evident along the 1D Cu_3S_3 chain. The presence of cuprophilic interactions is further substantiated by the characteristic absorption peak at 145 cm^{-1} observed in Raman spectroscopy as depicted in Fig. S5 (online) [29,41]. The manifestation of these interactions was hypothesized to enhance the carrier transport properties within the material [28,29].

Cu-HT was further characterized by a comprehensive spectroscopic method. Fourier-transform infrared (FT-IR) spectroscopy (Fig. 1d) confirmed the absence of the S–H stretching vibration peak, typically found at 2560 cm^{-1} , suggesting the effective coordination of sulfhydryl groups with copper ions in Cu-HT. Furthermore, the presence of a peak in the $3200\text{--}3600 \text{ cm}^{-1}$ region corresponds to hydroxyl groups, indicating the lack of coordination between hydroxyl and copper. X-ray photoelectron spectroscopy (XPS) analyses (Fig. 1e, f) further substantiated this coordination, with the S $2p_{3/2}$ peak at 162.5 eV , a signature of sulfur atoms bonded to copper [42,43]. Additionally, the Cu $2p_{3/2}$ spectrum showed a clear peak at 933.1 eV without significant satellite peaks, indicating a dominant monovalent copper oxidation state. The chemical environment of hydrogen atoms in the material was investigated through nuclear magnetic resonance (NMR) spectroscopy. The ligand in Cu-HT is consistent with HT (Fig. S6 online). Moreover, the elemental ratios closely approximate the chemical formula, suggesting that the sample has a very high degree of purity (Table S2 online). These characterization results are consistent with the simulated crystal structure of Cu-HT, further validating the simulated structure.

The electrical properties of Cu-HT were meticulously examined through the fabrication of devices utilizing this material. Four-electrode devices were prepared using photolithography technology to assess the conductivity of Cu-HT (Fig. 2a). Given the rectangular cross-section of Cu-HT nanorods (Fig. S7 online), conductivity (*G*) was calculated using the formula described in Supplementary materials (online). Across multiple device tests (Fig. S8 online), Cu-HT demonstrated remarkable conductivity, reaching up to $8.13 \times 10^{-3} \text{ S cm}^{-1}$ at room temperature, which is one of the highest values for coordination polymers with 1D structure [44–46]. Furthermore, variable-temperature conductivity measurements on Cu-HT nanorod device showed a positive correlation between electrical conductivity and temperature (Fig. 2c), indicating its typical semiconductor characteristics. The activation energy of Cu-HT is 0.11 eV (Fig. 2d).

The electronic band structure of Cu-HT was explored through spectral characterization. The UV–vis absorption spectrum analysis revealed that Cu-HT exhibits a bandgap of 2.04 eV (Fig. 3a). Utilizing the work function formula ($\varphi = 21.22 - E_{\text{cutoff}}$), the Fermi energy level of Cu-HT was estimated to be -4.66 eV (Fig. 3b). The ultraviolet photoelectron spectroscopy (UPS) valence band spectrum

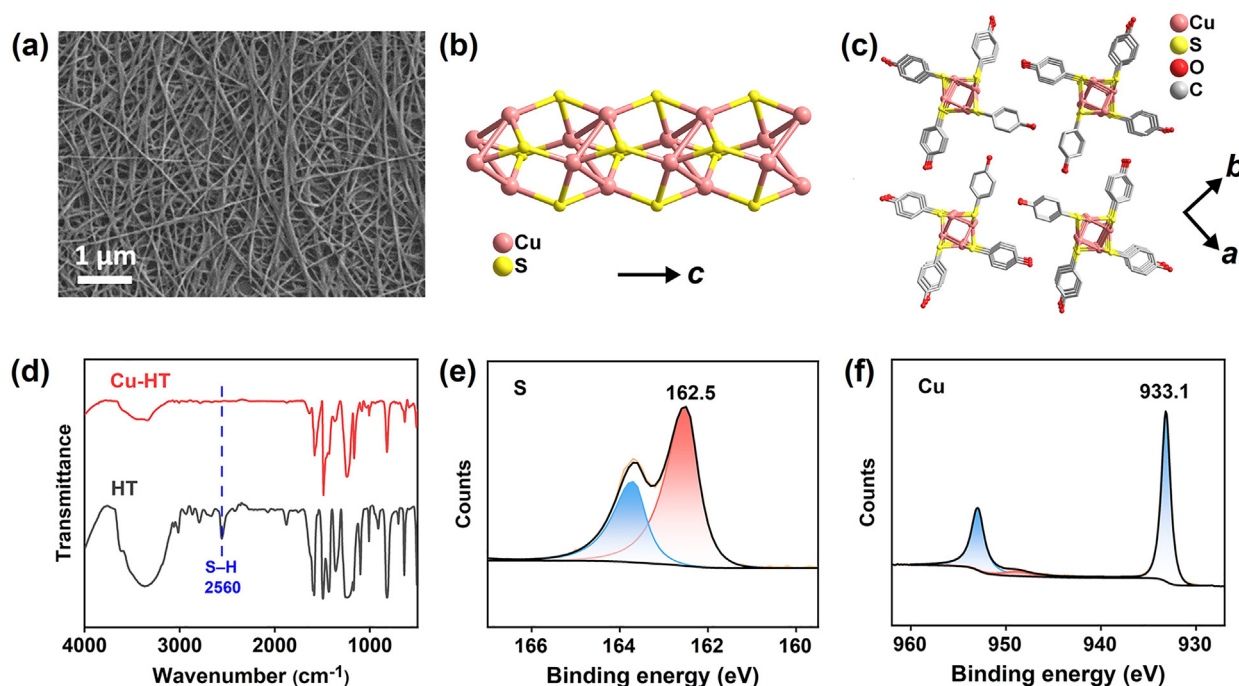


Fig. 1. (Color online) Structure and characterization of Cu-HT. (a) Scanning electron microscopy (SEM) image of Cu-HT. (b) (c) Structure viewed along the *c*-axis direction (benzene ring has been omitted) and *ab*-plane direction (hydrogen atoms has been omitted). (d) Fourier-transform infrared (FT-IR) spectrum of Cu-HT and HT ligand. (e, f) XPS of S and Cu.

Table 1
Synthesis condition for Cu-HT.

<i>T</i> (°C)	Stirring	Regulator	Diameter (nm)
30	✓	×	50–100
10	✓	×	100–200
30	✓	✓	300–500
30	×	✓	600–1400
10	×	✓	1000–2000

(Fig. 3c) shows that the valence band is positioned at 0.80 eV below the Fermi level (E_F). The combination of the valence band spectrum with optical band gap obtained from UV-vis spectroscopy shows that the E_F of Cu-HT is close to the valence band (Fig. 3d), suggesting that Cu-HT is a p-type semiconductor. Additional theoretical computations were harnessed to dissect the electronic structure and conductive trajectory of Cu-HT (Fig. 3e). The band structure diagram unveils an indirect band gap of 1.71 eV for Cu-HT, which is slightly lower than the value determined experimentally. This minor discrepancy is commonly observed in density functional theory (DFT) calculations because they tend to underestimate the band gap energy [47,48]. Moreover, the density of state (DOS) analysis shows significant contributions from the 3d orbitals of Cu and the 2p orbitals of sulfur near the valence band maximum (VBM). Fig. S9 (online) illustrates the frontier electron density of the VBM of Cu-HT, suggesting the strong orbital overlap and hybridization between Cu and S atoms to create effective hole transport channels of the Cu_3S_3 chains in Cu-HT [19,49]. Under the guidance of first principles and deformation potential theory, the theoretical hole mobility of Cu-HT is $48.6 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ (specific calculation details are found in Supplementary materials online).

The actual mobility of Cu-HT was evaluated through FET performance testing. Due to the poor contact with the substrate and the weak control of carriers in large-sized nanorods by field effects, Cu-HT nanorods with small diameters were used for the FET test

so the test results are close to the actual values. Multi-nanorod arrays were prepared for FETs through photolithography technology due to the aggregation between nanorods. FETs with a bottom gate/top contact structure were fabricated on a heavily doped p-type silicon substrate, with 300 nm silicon dioxide insulating layers and gold as source and drain electrodes (Fig. 4a). The SEM photos and specific preparation methods for FETs are summarized in Supplementary materials (online). The transfer characteristic curves (Fig. 4b) and output characteristic curves (Fig. 4c) of the Cu-HT FET demonstrate typical p-type conductivity, with an increase in drain-source current (I_{DS}) as the gate-source voltage (V_{GS}) decreases. Mobility was further calculated from the transfer characteristic curve because the saturation current was not attained in the transfer curves. Mobility was extracted from the linear region of transfer curves, as described in Supplementary materials (online). Across the multiple FET test, the hole mobility reaches $27.2 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$, with an average value of $18.7 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ and standard deviation of $\pm 4.6 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ (Fig. 4d). The parallel results are summarized in Fig. S10 (online). This mobility is one of the highest among 1D molecular-based materials, surpassing p-type organic semiconductors, such as DTT-DPP, PDVT-10, and DPPBTSPE. It even exceeds commonly used amorphous silicon and most 1D inorganic materials, such as CuO and single-wall carbon nanotubes (SWCNTs) [50]. The high hole mobility of Cu-HT may be attributed to its structural factors: (1) The formation of effective conductive channels through Cu and S atoms with high orbital overlap facilitates charge transport [18–20]; (2) the modification of the conductive channel surfaces with phenol functional groups possesses strong electron-donating properties, thereby reducing the hole injection barrier [24–26]; and (3) the presence of cuprophilic (Cu–Cu) interactions within the 1D channels of Cu-HT further enhances the carrier transport ability [28,29]. These structural features collectively contribute to the excellent hole mobility of Cu-HT, making it a strong contender for advanced semiconductor technologies.

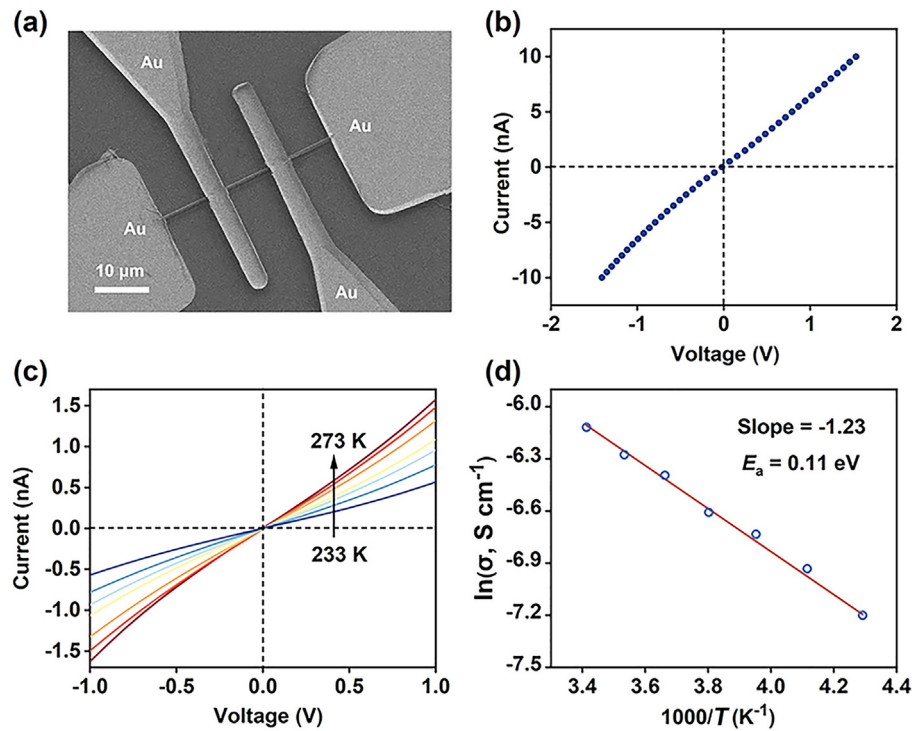


Fig. 2. (Color online) Electrical performance testing of a Cu-HT four-electrode device. (a) SEM image of four-electrode device for Cu-HT nanorod. (b) Current-voltage (I - V) curve of four electrodes. (c) Variable-temperature conductivity of Cu-HT. (d) Fitting of conductivity-temperature data to Arrhenius equation.

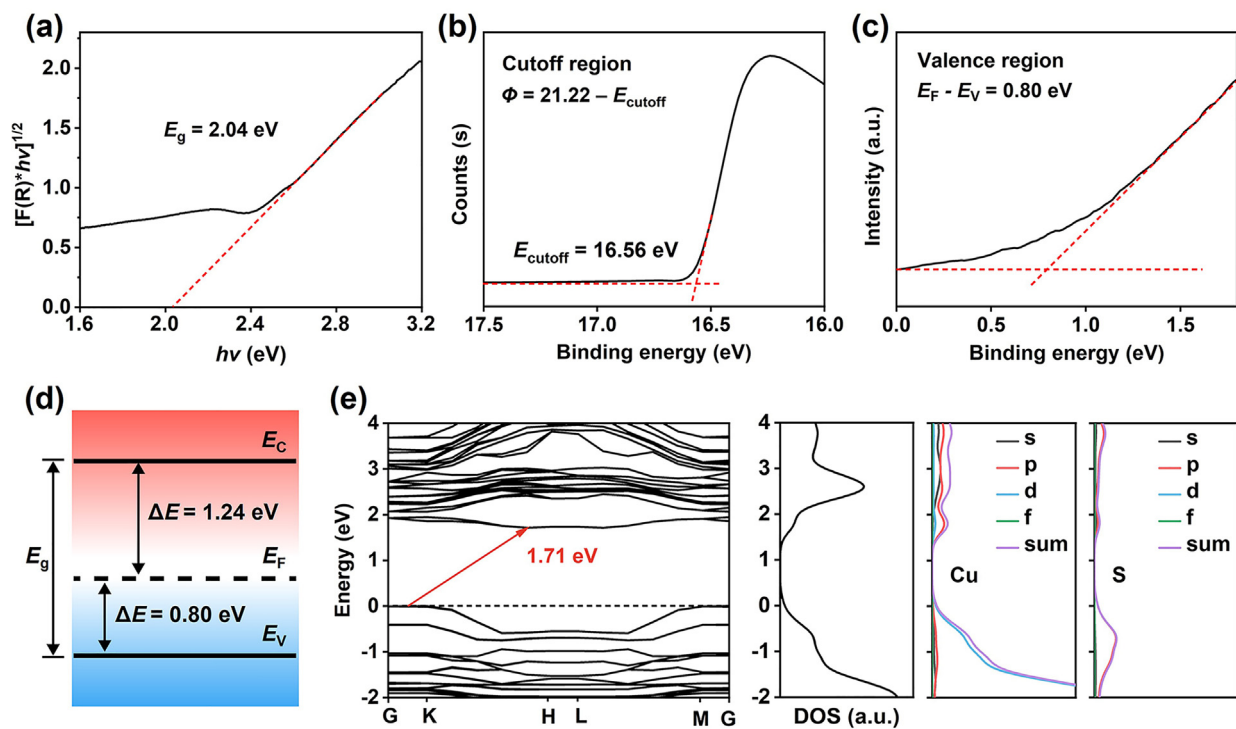


Fig. 3. (Color online) Spectroscopic characterization of the electronic band structure of Cu-HT. (a) UV-vis absorbance spectrum of Cu-HT. (b) Secondary electron cutoff of Cu-HT nanorods measured by Hel (energy of the photon $h\nu = -21.22 \text{ eV}$). (c) Valence band of Cu-HT with respect to the Fermi level ($E_F = 0$). (d) Band structure diagram of Cu-HT. (e) Theoretical band structure and density of states of Cu-HT.

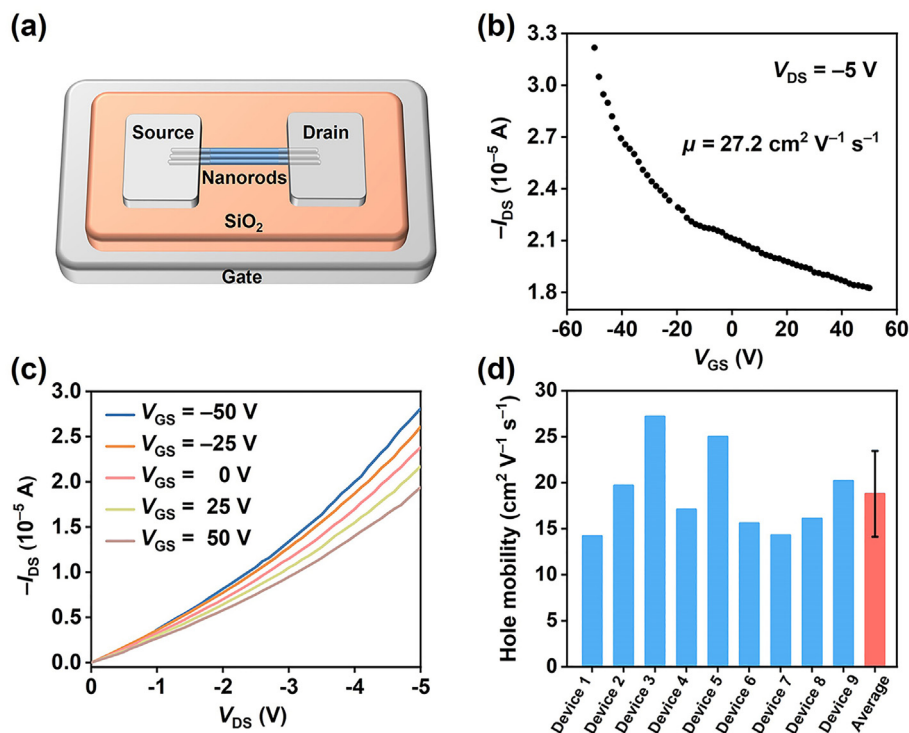


Fig. 4. (Color online) FET performance testing of Cu-HT. (a) Sketch map of FETs for Cu-HT. (b) The transfer characteristics curve ($V_{DS} = -5$ V). (c) Output characteristics curves and (d) statistical chart of hole mobility for FETs.

4. Conclusion

In conclusion, the crystal structure analysis reveals that Cu-HT is composed of Cu_3S_3 chains, which are enveloped by phenol functional groups known for their strong electron-donating capabilities. The material exhibits unique cuprophilic (Cu–Cu) interactions along the Cu_3S_3 chains. These features contribute to the exceptional electrical properties, as evidenced by the FETs that display typical p-type characteristics with a remarkable hole mobility of $27.2 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$. This value is not only among the highest reported for 1D molecule-based materials but also exceeds that of commonly used amorphous silicon as well as the majority of 1D inorganic materials. Furthermore, Cu-HT FETs were fabricated on silicon substrates through photolithography. The finding demonstrates the compatibility of Cu-HT with modern device manufacturing processes, indicating its potential for direct integration into established semiconductor fabrication workflows. Moreover, Cu-HT nanorods were transferred onto various substrates by drop-casting or using thermal release tape. These nanorods can conform to the curvature of flexible substrates, facilitating their easy incorporation into flexible electronic devices. The outstanding electrical performance demonstrates its potential applications in electronic devices. These findings will serve as a reference for the development of innovative electrical materials and offer a blueprint for the fabrication of advanced electrical materials that could revolutionize the microelectronics industry.

Conflict of interest

The authors declare that they have no conflict of interest.

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Author contributions

Chenhui Yu developed the synthesis method of Cu-HT, figured out the crystal structure as well as basic characterization of Cu-HT, and completed the initial draft of the article. Xinxu Zhu completed the testing of field-effect transistors and helped with subsequent revisions of the article. Kefeng Li proposed some innovative points in the experiment. Guane Wang and Gang Xu conceptualized and supervised the study, as well as acquired funding.

Appendix A. Supplementary materials

Supplementary materials to this article can be found online at <https://doi.org/10.1016/j.scib.2024.07.006>.

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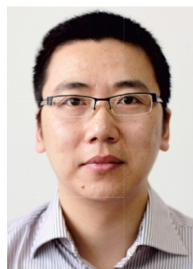
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