FelsnS4 Nanocrystals: A Ternary Metal Chalcogenide Material for Ambipolar Field-Effect Transistors

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An ambipolar channel layer material is required to realize the potential benefits of ambipolar complementary metal–oxide–semiconductor field-effect transistors, namely their compact and efficient nature, reduced reverse power dissipation, and possible scalability to highly integrated circuits. Here, a ternary metal chalcogenide nanocrystal material, FelsnS4, is introduced as a solution-processable ambipolar channel material for field-effect transistors (FETs). The highest occupied molecular orbital and the lowest unoccupied molecular orbital of the FelsnS4 nanocrystals are determined to be −5.2 and −3.75 eV, respectively, based upon cyclic voltammetry, X-ray photoelectron spectroscopy, and diffraction reflectance spectroscopy analyses. An ambipolar FelsnS4 FET is successfully fabricated with Au electrodes (EF = −5.1 eV), showing both electron mobility (14.96 cm² V⁻¹ s⁻¹) and hole mobility (9.15 cm² V⁻¹ s⁻¹) in a single channel layer, with an on/off current ratio of 10⁷. This suggests that FelsnS4 nanocrystals may be a promising alternative semiconducting material for next-generation integrated circuit development.

1. Introduction

For several decades, ambipolar field-effect transistors (FETs) have attracted considerable attention as a potential complementary metal–oxide–semiconductor (CMOS) transistor technology that could meet stringent application requirements for compactness and efficiency. Although conventional CMOS transistors provide lower power dissipation and higher density of logic functions on a chip for integrated circuits (ICs), their complicated device structure is accompanied by high cost and complex fabrication steps. Ambipolar FETs, which allow simultaneous electron and hole transport with simplified devices and fabrication processes, may be a next-generation alternative for CMOS circuits, avoiding the need to isolate two types of unipolar transistors from each other to prevent leakage current in conventional silicon-based devices. Therefore, there have been many efforts to demonstrate charge carrier ambipolarity in conducting channels as a means to realize the compact and simple device structure offered by ambipolar FETs.

To demonstrate ambipolar transport in a single channel layer, it is necessary for the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) levels of the semiconducting material to be suitably positioned relative to the work functions of the electrode materials. This can be achieved by choosing materials that have appropriate bandgap structures, doping to tune the bandgap, or combining both n- and p-type semiconducting materials to form the channel. In the trend toward more efficient device manufacturing and miniaturization of device structure, there is no doubt that the intrinsic bandgap structure of materials is most essential to characterize ambipolar transport behavior. In the same context, various materials have been applied to FETs to demonstrate ambipolar transport in simple device structure. For example, combined n- and p-type organic-based FETs display ambipolar transport with great potential for cost efficiency, large-scale production, light weight, and physical flexibility, but their low stability and mobility remain as challenges to overcome. FETs based upon carbon nanotubes, especially single-walled nanotubes (SWNTs), have shown ambipolar transport with high field-effect mobility (79 000 cm² V⁻¹ s⁻¹) and a high on/off ratio (10⁷). However, the challenges of purifying and separating SWNTs selectively from multiwalled nanotubes or graphene hinder their industrial application in ambipolar transistors because these impurities are metallic in character, promoting leakage in the off state and thereby worsening the on/off ratio considerably.
Additionally, inorganic nanocrystals (NCs) have emerged that exhibit electrical properties with great application potential such as wide libraries of bandgap tuning by means of doping, functionalization, and morphology and size adjustment. Moreover, because the solution process enables cost-effective fabrication, the application of NCs as FET channel layer materials has been widely studied, especially for NCs of cadmium and lead chalcogenides. Recently, various studies were performed to improve low charge transport ability by overcoming the poor contacts arising from the large interparticle spacing between NCs using strategies such as ligand exchange, Lewis base treatment, or building a superlattice structure. However, the fact that only lead chalcogenides have displayed ambipolar behavior, due to their intrinsic band structure, still hinders development of NC-based ambipolar FETs. Furthermore, there have been many efforts to achieve ambipolar transistors using lead chalcogenides that have included passivation, ligand exchange, and doping. However, the resulting devices still suffer from low mobility and poor stability under ambient conditions. To complicate matters further, lead is a well-known poison and its use hinders industrial applications. Therefore, development of new NC materials demonstrating ambipolar transport is highly desirable to fully realize the promise of compact and cost-effective IC based upon ambipolar FETs.

In the present work, we report the first demonstration of FeIn$_2$S$_4$ NCs as a new solution-processable ambipolar FET channel material. We designed a device structure that allows FeIn$_2$S$_4$ NCs to impart ambipolar transport properties, notably by ensuring suitable band edge positions relative to the Fermi level of a noble metal electrode material, namely Au (5.1 eV). The characteristics of the band edges of the as-synthesized FeIn$_2$S$_4$ NC were investigated by using X-ray photoelectron spectroscopy (XPS), cyclic voltammetry, and diffuse reflectance spectroscopy (DRS). Our results are expected to provide both electron and hole transport ability for ambipolar FETs. The resulting electrical characteristics will be discussed with regard to I–V curves from FeIn$_2$S$_4$ FETs.

2. Results and Discussion

2.1. Characterizations of FeIn$_2$S$_4$ Nanocrystals

FeIn$_2$S$_4$ NCs were prepared by means of a hydrothermal reaction method described in a previous study. As-synthesized nanoparticles were cleaved into smaller size via sonication with N-methyl-2-pyrrolidone (NMP), such that charge transport was not hindered due to large interparticle distance but a densely packed film was formed. To determine the crystal structure and phase of FeIn$_2$S$_4$ NCs, high-resolution transmission electron microscopy (HRTEM) images were acquired. The as-synthesized FeIn$_2$S$_4$ NCs were 500 nm in diameter. The characteristics of the band edges of the as-synthesized FeIn$_2$S$_4$ NC were investigated by using X-ray photoelectron spectroscopy (XPS), cyclic voltammetry, and diffuse reflectance spectroscopy (DRS). Our results are expected to provide both electron and hole transport ability for ambipolar FETs. The resulting electrical characteristics will be discussed with regard to I–V curves from FeIn$_2$S$_4$ FETs.
then were cleaved into single-crystalline petal-like NCs less than 100 nm in diameter (Figure 1b). Their atomic arrangement was preserved after cleavage into smaller NCs (Figure 1c and inset), with high crystallinity that was evidenced on selected area electron diffraction (SAED) patterns (Figure 1d). The spinel structure of FeIn$_2$S$_4$ NC is illustrated in Figure 1e. As explained above, tetrahedral and octahedral structures occupy each FeIn$_2$S$_4$ unit cell, each having different probabilities of containing Fe$^{3+}$ (green color in Figure 1e) and In$^{3+}$ ions (purple color in Figure 1e), which is supported by the high-resolution images obtained from the (220) plane of FeIn$_2$S$_4$ NCs.

The crystal structure and chemical bonding of the NCs were also investigated. X-ray diffraction (XRD) peaks can be indexed as spinel structures of FeIn$_2$S$_4$ (JCPDS Card No. 80-0608 shown in Figure S1 in the Supporting Information) with peaks at 14.3°, 23.48°, 27.54°, 28.8°, 33.38°, 43.88°, 48.02°, 50.4°, 56.16°, 59.76°, 66.96°, 70.18°, and 77° corresponding to the hkl planes of the (111), (220), (311), (222), (400), (511), (440), (531), (533), (444), (731), (800), and (822) phases ($a = 10.61$ Å), indicating high crystallinity and no impurities (Figure 2a). The chemical bonding of the compound was probed by means of XPS with the core levels of Fe 2p, In 3d, and S 2p, at respective binding energies of 702, 445, and 162 eV (Figure 2b–d). The Fe 2p binding energy peaks were of relatively low intensity (Figure 2d), suggesting relatively little bonding of iron compared with that of indium and sulfur ions. A likely explanation for this observation is that the FeIn$_2$S$_4$ NCs had a thiospinel structure, which can be described as AB$_2$S$_4$, whereas A, B, and S represent tetrahedral, octahedral structure and sulfur ions, respectively; in this unique crystal structure, the Fe$^{3+}$ ions would occupy the octahedral sites only, whereas the In$^{3+}$ ions would be equally distributed among the tetrahedral sites.[16]

2.2. Bandgap and Band Edge Investigation

DRS spectra were obtained to investigate the bandgap of the FeIn$_2$S$_4$ NC material. From the reflectance spectrum of FeIn$_2$S$_4$ NCs (Figure 3a), the optical absorption coefficient ($\alpha$) was obtained from the Kubelka–Munk equation as

$$F(R) = \alpha = (1 - R)^2 / 2R \tag{1}$$

where $R$ is the reflectance value from the sample.[17] To understand the optical bandgap of the FeIn$_2$S$_4$, the Kubelka–Munk function was used as

$$F(R) = A(h\nu - E_g) \tag{2}$$

where $E_g$ is the bandgap energy, $A$ is a constant representing the transition probability, and $p$ is a power index that is related to the optical absorption process; $p$ corresponds to allowed transitions, with a direct transition for $p = 1/2$ and an indirect transition for $p = 2$ (Figure 3b,c). Eventually, $E_g$ can be determined by extrapolating the linear regions of each plot using $E_{g,d} = 2.12$ eV for $p = 1/2$ and $E_{g,ind} = 1.67$ eV for $p = 2$.

Because the calculated result for the optical bandgap provides only the difference between the HOMO and LUMO, the redox potential of the FeIn$_2$S$_4$ NC material was determined to investigate the band structure, including the conduction and valence

![Figure 2. Structural and chemical characterization. a) XRD pattern of as-synthesized FeIn$_2$S$_4$ NCs. b–d) XPS core level spectra of b) In 3d$_{5/2}$, c) S 2p, and d) Fe 2p.](image-url)
band edges (Figure 3d). It has already been reported that the value of the optical bandgap shows good agreement with the quasiparticle gap, which can be obtained from dispersions of NCs.[18] Furthermore, redox potentials can provide information not only regarding the \( E_g \) of NCs but also regarding the position of the HOMO and LUMO edges through calibration versus the redox coupling of ferrocene (Fc/Fc\(^+\)) using the equations

\[
I_p = -4.8 - (E_m - 0.4245) \text{ eV} \tag{3}
\]

and

\[
EA = -4.8 - (E_{\text{red}} - 0.4245) \text{ eV} \tag{4}
\]

where \( I_p \) and \( EA \) are the ionization potential and electron affinity, respectively, and the 0.4245 eV term represents the half-wave potential of ferrocene (Figure S3, Supporting Information) relative to a reference electrode (0.01 m Ag/AgCl).[19] A cyclic voltammogram of monodisperse FeIn\(_2\)S\(_4\) NCs in a solvent of 0.1 m tetrabutylammonium hexafluorophosphate (TBAPF\(_6\)) in acetonitrile was obtained at a scan rate of 200 mV s\(^{-1}\), which showed an \( I_p \) and \( EA \) of −5.2535 and −3.7235 eV, respectively. In addition, considering the difference between \( I_p \) and \( EA \), the \( E_g \) of FeIn\(_2\)S\(_4\) NCs is 1.53 eV, which is closer to the optical bandgap of the indirect transition.

Photoelectron spectroscopy is also useful for determining the bandgap positions and Fermi level of semiconducting materials. In the present work, ultraviolet photoelectron spectroscopy (UPS) spectra were obtained to determine the absolute valence band position of FeIn\(_2\)S\(_4\), and XPS spectra were acquired to determine the difference between the Fermi level and the valence band of FeIn\(_2\)S\(_4\) (Figure 4).[20] Regarding band alignment in a device, to understand the charge carrier transport mechanism, it is important to establish the exact Fermi level of the semiconducting material. According to UPS spectra obtained using He I excitation (21.2 eV), the valence band position of FeIn\(_2\)S\(_4\) appeared to be around −5.2 eV, with a high-energy cutoff of 16 eV (Figure 4a). An XPS valence band maxima (VBM) spectrum showed that the difference between the \( E_F \) and VBM was around 0.7 eV (Figure 4b). As shown in XRD and XPS core level spectra of FeIn\(_2\)S\(_4\) NCs, no impurities were detected that play a role of unintended dopant. It is possible to assume that \( E_F \) would be positioned in the middle of the bandgap based on the Fermi–Dirac distribution theory, which is also well matched with the results of this study. Table 1 summarizes the results of each characterization of the FeIn\(_2\)S\(_4\) NCs. Since no impurities were evident in the XRD and XPS spectra, it is appropriate to assume that the Fermi level of FeIn\(_2\)S\(_4\) might be located in the middle of the bandgap.

Table 1 summarizes the results of each characterization of the FeIn\(_2\)S\(_4\) NCs. Since no impurities were evident in the XRD and XPS spectra, it is appropriate to assume that the Fermi level of FeIn\(_2\)S\(_4\) might be located in the middle of the bandgap. From the summarized results on the band structure of the FeIn\(_2\)S\(_4\) NCs (\( E_C = 5.25 \text{ eV} \) and \( E_V = 3.72 \text{ eV} \)), we can expect that, with most noble metal electrodes (\( E_F \) = 4.1–5.2 eV), they will show ambipolarity that can transport both electrons and holes. The ability of a transistor to operate in an ambipolar manner is determined by several factors, including the bandgaps of the semiconductor, the transport behavior of both carriers generated at the semiconductor/dielectric interface, and injection of both carriers through the interface between the metal electrodes.

![Figure 3. Bandgap and edge characterization of FeIn\(_2\)S\(_4\) thin films. a) Diffuse reflectance spectrum of the FeIn\(_2\)S\(_4\) NC film. b,c) Transformed Kubelka–Munk spectra of the FeIn\(_2\)S\(_4\) NC film: b) indirect transition and c) direct transition. d) Redox voltammograms of FeIn\(_2\)S\(_4\) NCs on a glassy carbon working electrode in a solvent of 0.1 m TBAPF\(_6\) in acetonitrile; scan rate: 200 mV s\(^{-1}\).](image-url)
The structure of FeIn\textsubscript{2}S\textsubscript{4} FET device is illustrated schematically in Figure 5a. The absence of a passivated organic surfactant that has different energy levels and low conductivity \cite{2} enhanced the transfer of charge carriers through the interparticles. In the fabrication procedure to obtain the FeIn\textsubscript{2}S\textsubscript{4} NCs, no organic surfactant was used as shown in XPS (Figure 2b–d). A high Schottky barrier junction current was observed (Figure 5e), which is frequently observed in ambipolar transistors.\cite{31} The high mobility obtained in Figure 5c,d can be explained by a surfactant effect. In the fabrication procedure to obtain the FeIn\textsubscript{2}S\textsubscript{4} NCs, no organic surfactant was used as shown in XPS (Figure 2b–d). The absence of a passivated organic surfactant that has different energy levels and low conductivity\cite{2} enhanced the transfer of charge carriers through the interparticles. To help explain the transport behavior between the interfaces of each of the building blocks during device operation, Figure 6 gives the schematic band alignment for each of the channel layers, namely the FeIn\textsubscript{2}S\textsubscript{4} NC film, the SiO\textsubscript{2} gate dielectric, the heavily boron-doped Si gate electrode, and the Au electrode. Figure 6a shows the flat band state, and Figure 6b shows the corresponding energy band diagram under equilibrium conditions. Under positive gate voltage, the electrons increase in concentration because they accumulate at the interface between the gate dielectric and the channel layer, which is accompanied by a shift in the bandgap to a higher potential energy (Figure 6c). Eventually, as the conduction band of the channel layer approaches the Fermi level of the source and drain electrodes, electrons can tunnel through the interface of the junction. Contrastingly, under a negative back gate voltage, because holes can accumulate at the interface between the channel layer and the gate dielectric, holes can travel through the interface and thus act as major carriers (Figure 6d). The electron mobility appears slightly higher than the hole mobility in the transfer curves because the middle of the bandgap of FeIn\textsubscript{2}S\textsubscript{4} is shifted slightly higher than the Fermi level of the Au electrodes.

\begin{equation}
\mu_{\text{sat}} = \frac{2L}{W} C \left( \frac{dI_D}{dV_C} \right) \quad (6)
\end{equation}

where \( W \) and \( L \) are the channel width and length, and \( C \) is the capacitance of the 100 nm SiO\textsubscript{2} (\( \approx 3.45 \times 10^{-8} \text{ F cm}^{-2} \)).\cite{10a} From the transfer curve, the electron and hole mobilities (\( V_{DS} = 1 \text{ V} \)) in the linear regime were 14.96 and 9.15 cm\textsuperscript{2} V\textsuperscript{-1} s\textsuperscript{-1}, and the mobilities (\( V_{DS} = 1 \text{ V} \)) in the saturation regime were 0.27 and 0.2 cm\textsuperscript{2} V\textsuperscript{-1} s\textsuperscript{-1}, respectively. The on/off current ratios were found to be 10\textsuperscript{5} for both electron and hole conduction. From these results, it is clear that the Fermi level of the Au was aligned close to the center of the gap between the HOMO and LUMO levels of FeIn\textsubscript{2}S\textsubscript{4} NCs, due to Fermi level pinning phenomenon. A high Schottky barrier junction current was observed (Figure 5e), which is frequently observed in ambipolar transistors.\cite{31} The high mobility obtained in Figure 5c,d can be explained by a surfactant effect. In the fabrication procedure to obtain the FeIn\textsubscript{2}S\textsubscript{4} NCs, no organic surfactant was used as shown in XPS (Figure 2b–d). The absence of a passivated organic surfactant that has different energy levels and low conductivity\cite{2} enhanced the transfer of charge carriers through the interparticles.

2.3. Electrical Characteristics of FeIn\textsubscript{2}S\textsubscript{4} FETs

The structure of FeIn\textsubscript{2}S\textsubscript{4} FET device is illustrated schematically in Figure 5a. Transfer (drain current vs gate voltage, \( I_D \) vs \( V_G \)) and output (\( I_D \) vs drain voltage \( V_D \)) characteristics were measured at room temperature (Figure 5b–e). V-shape transfer curves representing typical ambipolar transfer characteristics were observed. Both the electron and hole mobilities in the linear and saturation regimes were extracted using the equation

\begin{equation}
\mu_{\text{sat}} = \frac{L}{W} C \left( \frac{dI_D}{dV_C} \right) \quad (6)
\end{equation}

Table 1. Band energy characteristics of FeIn\textsubscript{2}S\textsubscript{4} NCs. The band energy characteristics are measured by means of diffuse reflectance spectroscopy, electrochemical measurements, and photoelectron spectroscopy. VBM, \( E_F \), CBM, and \( E_V \) represent the valence band maximum, Fermi level, conduction band maximum, and bandgap versus vacuum level, respectively.

| Method                        | VBM [eV] | \( E_F \) [eV] | CBM [eV] | \( E_V \) [eV] | \( \Delta E_g \) [eV] |
|-------------------------------|----------|----------------|----------|----------------|----------------------|
| Electrochemical measurement   | −5.25    | −3.72          | 1.53     |                |                      |
| Photoelectron spectroscopy    | −5.2     | −4.5           |          |                |                      |
| Diffuse reflectance spectrum  | 1.67 eV (indirect), 2.12 eV (direct) |                  |          |                |                      |
Figure 5. Device characteristics of FeIn$_2$S$_4$ NC FETs. a) Schematic illustration of FeIn$_2$S$_4$ FET. b) Transfer characteristics. c, d) Plots of $I_D$ and $I_D^{1/2}$ versus $V_G$ at constant $V_{DS} = 1$ V for ambipolar FETs assembled from FeIn$_2$S$_4$ NCs ($L = 25$ µm, $W = 50$ µm). e) Output characteristics: $I_D$ versus $V_{DS}$ for various $V_G$ for a hole-transport FET. Two different tunneling phenomena were distinguished in two voltage regions: i) direct tunneling (red dashed line) and ii) Fowler–Nordheim (FN) tunneling (black dashed line). Output characteristics for electron transport are given in Figure S5 (Supporting Information). f) Electrical characteristics were obtained for the same device after storage for two months under ambient conditions.

Figure 6. Energy band diagrams for the FeIn$_2$S$_4$ FET: a) flat band; b) equilibrium, $V_G = 0$; c) accumulation of electrons, $V_G > 0$; d) accumulation of holes, $V_G < 0$. Red arrows indicate the transfer of electrons and holes.
This result agrees with the bandgap characterizations obtained from cyclic voltammetry and photoelectron spectroscopy measurements.

In heterojunctions, especially metal–semiconductor junctions, triangular Schottky barriers inevitably form at the interface as a result of Fermi level pinning.[23] According to the Schottky–Mott rule, the height of the Schottky barrier is determined by the difference in energy states between the work function of the metal and the conduction band of the semiconductor, as expressed by the equation

\[
\Phi_{\text{BS}} = \Phi_{\text{m}} - X
\]

where \( \Phi_{\text{BS}} \) is the Schottky barrier height, \( \Phi_{\text{m}} \) is the work function of the metal, and \( X \) is the electron affinity of the semiconductor.[23] Based on this, the Schottky barrier height between FeIn\(_2\)S\(_4\) and Au is around 0.6–0.8 eV, which is not negligible for charge carrier transport. Through this barrier, carriers can tunnel through the interface by means of two phenomena: direct tunneling and Fowler–Nordheim (FN) tunneling.[24] Two significantly different slopes are observed in the curve of current versus drain voltage in two different voltage regimes (Figure 5e(i),(ii) and Figure S6 (Supporting Information)), so it can be assumed that there is a Schottky barrier at the interface between the channel layer and the electrodes, in good agreement with the characterization results for FeIn\(_2\)S\(_4\) NCs (Table 1).

Furthermore, the fabricated FET device showed high stability under ambient conditions for two months (Figure 5f) and also exhibited superior mobility and on/off ratio to lead with the characterization results for FeIn\(_2\)S\(_4\) NCs (Table 1).

3. Conclusion

A transition metal chalcogenide material of FeIn\(_2\)S\(_4\) NCs was synthesized, and various characterizations including DRS, cyclic voltammetry, XPS, and UPS were performed to demonstrate its feasibility for use as an ambipolar material with an appropriate bandgap and band edge levels for application in ambipolar FETs. The FeIn\(_2\)S\(_4\) NC FET device fabricated by means of solution processing showed ambipolar properties, high electron and hole mobilities, high on/off current ratio, and good stability even under ambient conditions. Owing to its cost-effective fabrication and ability to be used in ambipolar FETs for compact CMOS circuits, the FeIn\(_2\)S\(_4\) NC material reported herein appears promising as a next-generation semiconducting material.

4. Experimental Section

Materials: A previously reported procedure was followed to synthesize FeIn\(_2\)S\(_4\) microspheres.[15] Because the size and morphology of the resulting materials was not yet suitable for use in the FET channel layer, the microspheres were cleaved and isolated by means of lengthy sonication in the solution phase (Figure S2, Supporting Information).

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.
Keywords
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