Tunable superconductivity by electrochemical intercalation in TaS$_2$

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Abstract

The controllable manipulation of materials properties has always attracted broad interests since there exists the possibility to identify the relationship between different electronic states, and even discover new states. Here we report the electrochemical intercalation of cetyltrimethylammonium (CTA$^+$) into 2H-TaS$_2$. The single layer TaS$_2$ is intercalated with CTA$^+$ cations and the direction of CTA$^+$ is approximately perpendicular to the lamellar structure of TaS$_2$. With this facile and efficient method, the superconducting transition temperature ($T_c$) of pristine TaS$_2$ increases from 0.8 K to the maximum 3.7 K, showing a dome-like behavior. The Hall coefficient measurement indicates that the intercalated CTA$^+$ cations introduce electrons to this system. The present results demonstrate that electrochemical intercalation method is effective in tuning the materials properties and may pave the way for exploring new superconductors.

Introduction

The research of low-dimensional systems, especially the transition metal dichalcogenides (TMD), has always been a hot issue since there exist abundant phenomenon, such as the coexistence and competition of various collectively ordered electronic states [1—4]. Naturally, searching for a way to controllably manipulate these electronic states becomes significant. A common approach is chemical-doping, which has promoted the finding of high $T_c$ cuprates and iron-pnictides by suppressing the antiferromagnetism and spin density wave, respectively [5—7]. However, the limited manipulating potential and the structure complexity brought by elemental doping or replacement hinder the discovery of new physics. In contrast, the newly developed field-effect transistor (FET), especially the electric double-layer FET (EDLT) [8, 9] and solid ion conductor FET (SIC-FET) [10], has greatly overcome these disadvantages. For instance, high-temperature superconductivity could be achieved at high as 48 K in thin flakes of FeSe from less than 10 K for the pristine FeSe with the EDLT technology [8]. Moreover, the $T_c$ of FeSe could be enhanced to 46.6 K and an unexpected insulating state could be reached at the extremely overdoped regime via driving lithium ions into FeSe thin flakes with SIC-FET [10]. Nevertheless, the difficulties in fabrication and measurement of devices have limited more studies on low-dimensional system.

The electrochemical intercalation is a facile and efficient route to manipulate the structure and electronic states in van der Waals materials. The guest molecules or ions can be driven by electrochemical potential and then intercalated into the van der Waals gap. For instance, Cu ions could be intercalated into the topological insulator Bi$_2$Se$_3$ and eventually shows superconductivity at 3.6 K with electrochemical method [11]. Besides, the quality of electrochemical intercalated sample is much higher than the conventional melt growth ones. Recently, it is reported that a hybrid super-lattice of TiS$_2$ and organic cations is obtained by electrochemical intercalating molecules into TiS$_2$ [12]. This new hybrid material is an n-type flexible thermoelectric material whose thermoelectric figure of merit, ZT, increases by 4 times compared with the pristine TiS$_2$.

Tantalum disulphide (TaS$_2$) is a representative member of the TMD family. In TaS$_2$, the covalently bonded S-Ta-S planes stack upon each other and a variety of polytypic phases form from the different in-plane Ta coordination spheres and the stacking periodicity [13]. Among all the polytypic phases, the 1T and 2H polytypes
are the most studied, where the 1T structure is in octahedral coordination and the 2H structure is in trigonal prismatic coordination. Although they have been extensively studied in the 1970s [14, 15], the two polytypic phases again attract much attention due to the fact that they may act as the ideal role for the investigation of competing orders, namely, charge density waves (CDW), superconductivity and hidden order [16, 17]. In this scenario, the controllable manipulation of these orders might bring in new insight in understanding these phenomena. Here we report the controllable electrochemical intercalation into 2H–TaS2 with cetyltrimethylammonium (CTA+). The single layer TaS2 is intercalated with CTA+ cations and form this hybrid material. The direction of CTA+ is approximately perpendicular to the lamellar structure of TaS2. By intercalating CTA+ cations, the superconducting transition temperature (Tc) of pristine TaS2 increased from 0.8 K to the maximum 3.7 K. Meanwhile, with the increase amount of intercalated CTA+, the superconducting transition temperature firstly increases and then decreases, showing a dome-like behavior. Moreover, the Hall coefficient measurement indicates that the intercalated CTA+ cations introduce electrons to this system. The upper critical field of this hybrid material is estimated to be 0.63 T from magneto-resistance measurement.

**Experiment**

The TaS2(CTA+)x is synthesized via electrochemical intercalating cetyltrimethylammonium bromide ((C16H33)N(CH3)3Br, abbreviated as CTAB) into 2H–TaS2 single crystal. The pristine TaS2 single crystal is synthesized by chemical vapor transport method. High purity powders of elements Ta and S are stoichiometric mixed and thoroughly grounded. The mixture is then sealed under vacuum in a quartz tube with a small quantity of iodine (5 mg cm−2). The tube is placed into a two-zone horizontal furnace with the hot end of 875 °C and the cold end of 750 °C. High-quality plate-like 2H–TaS2 single crystals are obtained. Before the electrochemical intercalation procedure, those 2H–TaS2 single crystals are washed with ethanol and cut into rectangular pieces. Then, they are pressed onto an indium plate and acted as the working electrode. A 0.5 mm thick carbon cloth is used both as the counter electrode and reference electrode. For the intercalation, a supersaturated solution of CTAB powder (99.5%) dissolved in N-Methyl-2-purrolidone (NMP) is used. In order to realize a uniform intercalation, we applies a constant current of 2 μA to control the electrochemical intercalation. The samples with different x in TaS2(CTA+)x are acquired by controlling the time of galvanostatic discharge process. The whole intercalation process is controlled by Lanhe testing system. The electrochemical reaction can be described by two reaction equation

\[
\text{Br}^- \rightarrow 1/2\text{Br}_2 + e^- ,
\]

\[
\text{TaS}_2 + x(\text{CH}_3)\text{NC}_6\text{H}_{13}^+ + xe^- \rightarrow (\text{TaS}_2)_x[(\text{CH}_3)\text{NC}_6\text{H}_{13}]^x .
\]

The structure of TaS2(CTA+)x is characterized by a powder x-ray diffractometer (SmartLab-9, Rigaku Corp.) with CuKα radiation and a fixed graphite monochromator. The HRTEM images are obtained from a Talos F200X microscope with the operating voltage at 200 KV. The resistivity and Hall measurement is carried out by physical properties measurement system with standard four-terminal method. The low temperature (<2 K) measurement is conducted by a dilution refrigerator. The magnetic susceptibility was measured by a SQUID magnetometer (Quantum Design MPMS-5).

**Results and Discussion**

Figure 1(a) shows the x-ray diffraction pattern for TaS2(CTA+)x with x = 0, 0.1, 0.4, 0.9. The intercalated single crystals still maintain the plate-like morphology yet much thicker. The intercalated amount of CTA+, namely x in TaS2(CTA+)x, is calculated based on the theoretical specific charge capacity for full intercalation of 109 mAh g−1. Hence, the x in TaS2(CTA+)x, could be determined by the duration of galvanostatic discharge process. This method is also used in determination of Li content in electrochemical intercalated Li2MoS2 and Li,NbSe2 [18, 19]. The pristine 2H–TaS2 bears a space group of P63/mmc with the c-axis of 12.08 Å. By intercalating 0.1 CTA+ cation, the c-axis increased to 32.15 Å from 12.08 Å. Due to the fact that a single unit cell of 2H–TaS2 is formed by two individual layers (figure 1(b)), the height of a single layer of 2H–TaS2 is proximately 6.04 Å. Moreover, the c-axis of CTA+ is about 26 Å. The c-axis value of intercalated compounds happens to be the sum of one single layer of 2H–TaS2 and 1 crystalline CTA+ oriented in c-axis. Considering the fact that one end of CTA+ is negatively charged and may keep a distance with the sulfur-layer of TaS2, the CTA+ chain may incline a little. A similar stacking sequence is also observed in CTA+ intercalated V2O5 [20, 21]. Based on the above analysis, we propose a possible structure model, which is shown in figure 1(b). The single layer TaS2 is intercalated with CTA+ cations and the direction of CTA+ is approximately perpendicular to the lamellar structure of TaS2. However, due to the poor crystalline quality of our intercalated compounds, the proposed structure model is one of all the reasonable structures and further investigation is needed. Furthermore, with the
increase of the amount of intercalated CTA\(^+\), the distance of c-axis remains almost unchanged. This phenomena is also observed in the CTA\(^+\) intercalated clay in which there is no obvious change in the c-axis with increase of the amount of intercalated CTA\(^+\) in a range \([22]\). It is also worth noting that the diffraction peaks become much more weak and broad with more intercalation. When the x in TaS\(_2\)(CTA\(^+\))\(_x\) reaches 1, the diffraction peaks become too weak to observe, which could be ascribed to the poor crystalline quality after intercalating too many CTA\(^+\) cations.

Figure 2 shows the cross-section HRTEM images of TaS\(_2\)(CTA\(^+\))\(_x\) and pristine 2H–TaS\(_2\). The low magnification cross sectional images (figures 2(a) and (c)) clearly demonstrate the structure evolution before and after intercalation. From the high magnification images (figures 2(b) and (d)), it can be seen that the interlayer distance increases from 0.6 nm (monolayer 2H–TaS\(_2\)) to 3.24 nm, which is in good agreement with the previous XRD result and support the structure model we proposed. Besides, the layers in intercalated TaS\(_2\)(CTA\(^+\))\(_x\) are distorted and forming a wavy structure, which is also observed in TiS\(_2\) related materials \([12]\). This is mainly because the organic molecules intercalated materials become soft. Moreover, a more clear image is difficult to obtain due to the instability of the intercalated TaS\(_2\)(CTA\(^+\))\(_x\).

The temperature dependent magnetic susceptibility of all the samples is measured from 1.8 to 5 K, as shown in figure 3. Figures 3(a)–(d) show the typical M–T curves (Zero-field cooling and field cooling) for TaS\(_2\)(CTA\(^+\))\(_x\) with x = 0.1, 0.5, 0.6 and 0.9, respectively. Figure 3(e) shows the enlarged view of superconducting transition from magnetic susceptibility measurement as well as the criterion used to determine the T\(_c\). With the increase of the amount of CTA\(^+\) cations, the T\(_c\) initially increases rapidly. When the x is 0.6, the T\(_c\) reaches a maximum at 3.7 K. Then, the T\(_c\) gradually decreases and remains nearly unchanged, suggesting that the intercalation keeps saturation. The superconducting phase diagram of TaS\(_2\)(CTA\(^+\))\(_x\), is shown in figure 3(f).

The resistivity and Hall measurements are carried out for TaS\(_2\)(CTA\(^+\))\(_x\). The results are shown in figure 4. For pristine 2H–TaS\(_2\), it shows a charge-density-wave related phase transition accompanied by a kink in resistivity at 70 K. The superconductivity emerges at \(\sim\)0.8 K, which is in accordance with previous report \([23]\).
The temperature dependent resistivity for TaS$_2$($\text{CTA}^+$)$_x$, is also measured, as shown in figure 4(a). For the three samples, the CDW-like drop is completely vanished in the temperature range of 2–300 K. It indicates the intercalated CTA$^+$ cations destroy CDW order. Thus, in this system, there may exist a competition between CDW order and superconductivity. For the intercalated TaS$_2$($\text{CTA}^+$)$_x$, the resistivity $\rho$ decreases smoothly and then the superconducting transition occurs. The $T_c^{\text{zero}}$ observed in resistivity is in accordance with the value from magnetic susceptibility. The samples with more intercalated CTA$^+$ become so loose that it is difficult to obtain a good electrical contact. Figure 4(b) shows the Hall measurement of pristine TaS$_2$ and intercalated TaS$_2$($\text{CTA}^+$)$_{0.4}$. The temperature dependent Hall coefficient ($R_{H}$) of pristine TaS$_2$ is positive and nearly temperature-independent above the CDW temperature, then rapidly decreases and changes its sign below the CDW temperature. Such behavior is in accordance with previous report [24]. In 2H–TaS$_2$, there exists two kinds of charge carriers, electrons and holes. The two kinds of charge carriers dominate the transport behavior in different temperature range. However, for the intercalated TaS$_2$($\text{CTA}^+$)$_{0.4}$, the temperature dependent $R_{H}$ is distinctly different from that of pristine TaS$_2$. $R_{H}$ decreases gradually with decreasing temperature and remains negative throughout the whole temperature range. It indicates that the dominant carriers are electron type. This sign change of $R_{H}$ clearly indicates that the intercalated CTA$^+$ introduces electron into TaS$_2$. A similar case could also be found in Cu doped TaS$_2$ [25]. It is also worth noting that at the temperature points we measured for the all three samples, the field dependent Hall resistivity shows quite good linear dependence. Thus, we roughly estimate the carrier density in a simple way by a single-band model. Thus, utilizing this electrochemical intercalation method, a facile and effective way is realized to controllably tune the carrier density of 2H–TaS$_2$, and consequently to change the superconducting transition temperature. Figure 4(c) shows the resistivity of TaS$_2$($\text{CTA}^+$)$_{0.1}$ under different magnetic fields. With 

Figure 2. The cross-section high-resolution TEM images of TaS$_2$($\text{CTA}^+$)$_x$. (a) The low magnification image of TaS$_2$($\text{CTA}^+$)$_{0.4}$, showing a wavy structure. (b) The high magnification image of TaS$_2$($\text{CTA}^+$)$_{0.4}$. The interlayer spacing is 3.2 nm, being in accordance with x-ray diffraction result. (c) The low magnification image of 2H–TaS$_2$. (d) The high magnification image of 2H–TaS$_2$. The interlayer spacing is 0.6 nm, corresponding to the monolayer height of 2H–TaS$_2$. New J. Phys. 20 (2018) 023014 N Z Wang et al
the increase of magnetic field, the superconducting transition temperature is gradually suppressed and the transition is broadened. However, although the organic cations intercalated into the van der Waals gap of TaS$_2$, the resistivity behavior does not resemble the features such as field-induced thermally-activated flux-flow or vortex liquid which are observed in some other quasi two-dimensional systems, like (Li, Fe)$_2$OHFeSe, SmFeAsO$_{0.85}$ and cuprates [26–28]. Figure 4(d) shows the temperature dependent upper critical field $H_{c2}(T)$ of TaS$_2$(CTA$^+$)$_{0.1}$. The $H_{c2}(T)$ of TaS$_2$(CTA$^+$)$_{0.1}$ has a positive curvature. This is the characteristic of two-band clean-limit type-II superconductors and common in many layered superconductors, like WTe$_2$ under high pressure, MgB$_2$ and YNi$_2$B$_2$C [29–31]. Due to this theory, our experimental data $H_{c2}(T)$ can be well fitted by the equation $H_{c2}(T) = H_{c2}^0(1 - T/T_c)^{2+\alpha}$ [31], where the $H_{c2}(0)$ is estimated to be 0.63 T and $\alpha$ is 0.19. This value is higher than the reported upper critical field of 0.11 T for the pristine 2H–TaS$_2$ [32]. It is also worth noting that our estimated $H_{c2}(0)$ is less than the BCS weak-coupling Pauli limit.

The pristine 2H–TaS$_2$ usually exhibits a charge-density-wave transition accompanied by a kink in resistivity at around 70 K, which could be observed in figure 4(a). In contrast, the CDW-like kink in resistivity is completely absent in the whole temperature for intercalated TaS$_2$(CTA$^+$)$_x$. For the intercalated TaS$_2$(CTA$^+$)$_x$, the interlayer spacing is largely expanded, which could increase the two-dimensional anisotropy and in part maintain even enhance the CDW state. However, the reduced dimensionality could also suppress the long range order and enhance the fluctuation effect, which would lower the CDW temperature [33]. Besides, the intercalated CTA$^+$ cations would transfer charge to the TaS$_2$ layer and may enhance the three-dimensionality of TaS$_2$. The superconducting transition temperature is gradually suppressed and the transition is broadened. However, although the organic cations intercalated into the van der Waals gap of TaS$_2$, the resistivity behavior does not resemble the features such as field-induced thermally-activated flux-flow or vortex liquid which are observed in some other quasi two-dimensional systems, like (Li, Fe)$_2$OHFeSe, SmFeAsO$_{0.85}$ and cuprates [26–28]. Figure 4(d) shows the temperature dependent upper critical field $H_{c2}(T)$ of TaS$_2$(CTA$^+$)$_{0.1}$. The $H_{c2}(T)$ of TaS$_2$(CTA$^+$)$_{0.1}$ has a positive curvature. This is the characteristic of two-band clean-limit type-II superconductors and common in many layered superconductors, like WTe$_2$ under high pressure, MgB$_2$ and YNi$_2$B$_2$C [29–31]. Due to this theory, our experimental data $H_{c2}(T)$ can be well fitted by the equation $H_{c2}(T) = H_{c2}^0(1 - T/T_c)^{2+\alpha}$ [31], where the $H_{c2}(0)$ is estimated to be 0.63 T and $\alpha$ is 0.19. This value is higher than the reported upper critical field of 0.11 T for the pristine 2H–TaS$_2$ [32]. It is also worth noting that our estimated $H_{c2}(0)$ is less than the BCS weak-coupling Pauli limit.

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the system, which might suppress the CDW order. Thus, the collective effects mentioned above might lead to the elimination of CDW order in the intercalated TaS$_2$(CTA$^+$)$_x$. Moreover, accompanying with the intercalation process, the charge carrier concentration close to the superconducting transition temperature as well as $T_c$ is gradually increasing, which suggests that the enhancement of $T_c$ is strongly correlated with the improvement of carrier concentration and confirms the electron transfer from CTA$^+$ to TaS$_2$. Besides, the resistivities measured with different CTA$^+$ concentration do not show a systematic trend in absolute magnitudes. The reason might be that the intercalated samples are loose so that the thickness of samples could not be measured accurately, which leads to the deviation between calculated resistivity and the real one.

Recently, it has been reported the superconducting transition temperature and upper critical field would increase for 2H-TaS$_2$ as the layers are thinned down, which is attributed to the enhancement of effective electron–phonon coupling constant with reducing dimensionality [34]. In our case, although the interlayer spacing is greatly increased with intercalating organic cations, the measurement of resistivity under magnetic field implies TaS$_2$(CTA$^+$)$_{0.1}$ does not resemble some features observed in other quasi two-dimensional superconductors. Thus, the enhancement of superconductivity in TaS$_2$(CTA$^+$)$_{0.1}$ may be mainly caused by the charge transfer from the intercalated CTA$^+$ cations instead of the reduced dimensionality.

Figure 4. The resistivity and Hall coefficient of TaS$_2$(CTA$^+$)$_x$. (a) Temperature dependent resistivity for TaS$_2$(CTA$^+$)$_x$ ($x = 0, 0.1, 0.2, 0.3$, respectively). The inset of figure 4(a) shows the enlarged view. In order to clarify the transition, the resistivity of pristine 2H-TaS$_2$ in the inset of figure 4(a) is multiplied by three times. (b) The temperature dependent Hall coefficient for 2H-TaS$_2$ and TaS$_2$(CTA$^+$)$_x$ ($x = 0, 0.1, 0.2$). The inset shows the field dependent Hall resistance of TaS$_2$(CTA$^+$)$_{0.1}$, which shows good linear dependence. The Hall coefficients are measured from $-9$ to $+9$ T to eliminate any magnetoresistive due to the misalignment of the voltage contacts. (c) The temperature dependent resistivity of TaS$_2$(CTA$^+$)$_{0.1}$ under different magnetic field. (d) The estimation of upper critical field of TaS$_2$(CTA$^+$)$_{0.1}$.
Conclusions

In conclusion, using the electrochemical intercalation method, the CTA$^+$ cations are controllably intercalated into 2H-TaS$_2$. The single layer TaS$_2$ is intercalated with CTA$^+$ cations and forms this hybrid material. The direction of CTA$^+$ is approximately perpendicular to the lamellar structure of TaS$_2$. The superconducting transition temperature is enhanced by 0.8 K (pristine 2H-TaS$_2$) to a maximum of 3.7 K. The phase diagram of TaS$_2$(CTA$^+$), system is mapped out and shows a dome-like behavior. Moreover, the Hall coefficient measurement indicates that the intercalated CTA$^+$ introduce electrons to TaS$_2$ layers. Finally, our studies demonstrate that the electrochemical intercalation method is a facile and effective way to controllably manipulate the electronic properties of different two-dimensional materials. In particular, this method can open a new way to search for new high-temperature superconductors.

Acknowledgments

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