Diluted magnetic semiconductors based on Sb2ÀxVxTe3 „0.01îxî0.03.

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Diluted magnetic semiconductors based on $\text{Sb}_2-x\text{V}_x\text{Te}_3$ ($0.01 \leq x \leq 0.03$)

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We report on a diluted magnetic semiconductor based on the $\text{Sb}_2\text{Te}_3$ tetradymite structure doped with very low concentrations of vanadium (1–3 at. %). The anomalous transport behavior and robust magnetic hysteresis loops observed in magnetotransport and magnetic measurements are experimental manifestations of the ferromagnetic state in these materials. The $p$-$d$ exchange between holes and vanadium 3$d$ spins is estimated from the behavior of the magnetoresistance. A Curie temperature of at least 22 K is observed for $\text{Sb}_{1-y}\text{V}_{0.03}\text{Te}_3$. This discovery offers possibilities for exploring magnetic properties of other tetradymite structure semiconductors doped with a wide range of 3$d$ transition metals.

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I. INTRODUCTION

Since the first studies of the coexistence of ferromagnetism and semiconducting properties in Eu chalcogenides and semiconducting spinels,$^1$ the rich interplay between magnetic cooperative phenomena and semiconducting properties has received considerable attention. Diluted magnetic semiconductors (DMS’s), which display this behavior, are classical semiconductors where a controlled fraction of the nonmagnetic cation is substituted by transition-metal or rare-earth ions. The most extensively studied DMS’s are based on the II-VI or III-V ternary compounds where Mn$^{2+}$ ions serve as the magnetic impurity,$^2$ and ferromagnetic interaction among localized Mn spins has been seen in thin films of low-temperature molecular beam epitaxy (MBE)-grown $p$-type $\text{InAs}$ and $\text{GaAs}$$^{3–5}$. These DMS’s have tetrahedral bonds and crystallize in either the zinc-blende or wurtzite structure, and their magnetic properties are intimately tied to the presence of the manganese ions. Our diluted magnetic semiconductor is based on $\text{Sb}_2\text{Te}_3$ and differs from the existing DMS’s in two important respects: it crystallizes with an anisotropic tetradymite-type structure with atoms in an octahedral coordination; and the magnetic ion is not manganese but vanadium.

The parent crystal, $\text{Sb}_2\text{Te}_3$, is a narrow-band-gap semiconductor with tetradymite structure (space group $R\bar{3}m$ –$D_{3d}$) that belongs to the family of compounds with the form $A^2B^3_3$ (where $A = \text{Bi}$, $\text{Sb}$ and $B = \text{Se}$, Te). The crystal lattice of $\text{Sb}_2\text{Te}_3$ (see Fig. 1) consists of repeated groups of atomic layers, $\text{Te}^{(2)}$-$\text{Sb}$-$\text{Te}^{(1)}$-$\text{Sb}$-$\text{Te}^{(2)}$, oriented perpendicular to the trigonal $c$ axis, with primarily ionic and covalent bonding within the layers ($\text{Te}^{(1)}$-$\text{Sb}$ and $\text{Sb}$-$\text{Te}^{(2)}$), and van der Waals bonding between the Te sheets forming the double layers ($\text{Te}^{(2)}$-$\text{Te}^{(2)}$).$^6$ This interesting crystal structure results in anisotropic transport$^7$ and optical properties$^8$ and this class of materials forms the backbone of the present-day thermoelectric cooling devices.$^7$ $\text{Sb}_2\text{Te}_3$ is diamagnetic, just as the other pure $A^2B^3_3$ tetradymite semiconductors,$^9$ and little is known about the magnetic properties upon doping the structure.

In this work, we report on an observation of a ferromagnetic state that sets in at low temperatures when a minute amount of vanadium impurity is introduced into the $\text{Sb}_2\text{Te}_3$ matrix. Very recently, a similar material, $\text{Bi}_{2-x}\text{Fe}_x\text{Te}_3$ was identified as a DMS with a ferromagnetic transition temperature of up to 12 K for $x = 0.08$$^{10}$ In our structure, which contains a lower concentration of magnetic impurity (vanadium), the magnetic ordering effect appears to be considerably stronger than in the Fe-doped compound. These results bring forth a different semiconducting system that undergoes a ferromagnetic transition at low temperatures and as such broadens the scope of the existing diluted magnetic semiconductors.

FIG. 1. Atomic layers in the $\text{Sb}_2\text{Te}_3$ crystal structure. Dashed lines indicate van der Waals gaps separating five atomic layer lamella. The octahedral coordination is highlighted for a $\text{Te}^{(1)}$ atom.
II. EXPERIMENT

Single crystals of Sb$_{2-x}$V$_x$Te$_3$ with $x=0, 0.01, 0.02, 0.03$ (nominal) were synthesized using a modified Bridgman method. Bar shaped specimens for transport and magnetic studies were cut with a spark erosion machine to typical dimensions of $1.5 \times 2.5 \times 5.5$ mm$^3$ where the first dimension is parallel to the $c$ axis. Preparation of samples with their long edge parallel to the $c$ axis is exceedingly difficult because the structure cleaves very easily along the van der Waals–bonded Te double layers. X-ray lattice parameters were determined from powders with the aid of a Scintag powder diffractometer. Trends in the $c$ lattice parameter were also verified using thin single-crystal strata that were cleaved from larger specimens. The results, given in Table I, show that both $a$ and $c$ decrease monotonically upon addition of vanadium. Analysis of the composition was carried out by both atomic absorption spectroscopy and electron microprobe analysis (EPMA) and actual vanadium concentrations found to be close to the nominal values. No secondary phases were detected.

Galvanomagnetic measurements were carried out over the temperature range 2–300 K using a low-frequency (16 Hz) a.c. technique. Ohmic contacts to the samples were made with fine copper wires attached with care using a tiny amount of silver paint or indium solder. Magnetic studies were made in a superconducting quantum interference device (SQUID) magnetometer equipped with a 5.5-T superconducting solenoid and the data were corrected for a minor contribution arising from the small plastic sample holder.

III. RESULTS AND DISCUSSION

Anomalies in the low-temperature transport properties of Sb$_{2-x}$V$_x$Te$_3$ provided the initial hint of the developing long-range magnetic order. Temperature dependences of the in-plane ($LLc$) resistivity $\rho$ and Hall coefficient $R_H$ are shown in Figs. 2 and 3, respectively. Resistivity of the pure Sb$_2$Te$_3$ sample increases linearly with temperature over the entire temperature range indicating degenerate Fermi gas carrier transport. The Hall coefficient $R_H$ was obtained in a 10-kG magnetic field oriented parallel to the $c$ axis, and is positive with a modest temperature dependence for Sb$_2$Te$_3$. The room-temperature carrier (hole) concentration for Sb$_2$Te$_3$ is $1.0 \times 10^{20}$ cm$^{-3}$ due to native defects, typical for this compound. Upon addition of vanadium, the resistivity increases but remains metallic, while the values of the carrier concentration change very little. Room-temperature infrared reflectivity measurements in the plasma resonance frequency region (not shown here) corroborate this behavior.

<table>
<thead>
<tr>
<th>Nominal $x$ (EPMA)</th>
<th>$N_V$ (cm$^{-3}$)</th>
<th>$a$ (Å)</th>
<th>$c$ (Å)</th>
<th>$p$ (cm$^{-3}$)</th>
<th>$\rho$ (m$\Omega$ cm)</th>
<th>$\mu_H$ (cm$^2$/V s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>4.263(1)</td>
<td>30.445(6)</td>
<td>$1.0 \times 10^{20}$</td>
<td>0.21</td>
<td>297</td>
</tr>
<tr>
<td>0.01 (0.010±0.002)</td>
<td>6.3×10$^{19}$</td>
<td>4.260(8)</td>
<td>30.441(1)</td>
<td>$9.4 \times 10^{19}$</td>
<td>0.44</td>
<td>152</td>
</tr>
<tr>
<td>0.02 (0.016±0.002)</td>
<td>$1.0 \times 10^{20}$</td>
<td>4.260(3)</td>
<td>30.432(4)</td>
<td>$9.3 \times 10^{19}$</td>
<td>0.66</td>
<td>103</td>
</tr>
<tr>
<td>0.03 (0.026±0.002)</td>
<td>$1.6 \times 10^{20}$</td>
<td>4.259(0)</td>
<td>30.433(8)</td>
<td>$9.8 \times 10^{19}$</td>
<td>0.98</td>
<td>65</td>
</tr>
</tbody>
</table>
temperature properties are summarized in Table I. Error in the Hall hole concentrations is approximately 5%.

Below 30 K the presence of vanadium leads to a peak in both \( \rho \) and \( R_H \) that moves to higher temperature as the vanadium content increases. Magnetic semiconductors commonly exhibit a maximum in \( \rho \) near their Curie temperature \( T_C \). This critical behavior arises due to scattering of carriers by magnetic spin fluctuations via the exchange interaction. The presence of a magnetic field oriented parallel to the \( c \) axis suppresses this peak, as is seen by considering the transverse magnetoresistance data in Fig. 4. Rapid decrease in resistance and large negative magnetoresistance below \( T_C \) are a consequence of significantly reduced spin-disorder scattering. Above 50 K, the magnetoresistance is positive, has a parabolic dependence on temperature as is expected from Lorentz force effects, and the magnitude of \( \Delta \rho / \rho \) is less than 1% at 50 K.

Spin-disorder scattering \( \rho_s \) can be analyzed by a model proposed by Kasuya,\(^\text{13}\) as has been done in other DMS systems [e.g., (Ga, Mn) As (Ref. 14)] which has the form

\[
\rho_s = 2 \pi \frac{k_F}{e} \frac{m^2 J_{pd}^2}{h^3} N_V [S(S + 1) - (\langle S \rangle)^2],
\]

where \( k_F \) is the Fermi wave number, \( e \) is the elementary charge, \( m \) is the carrier effective mass, \( h \) is Planck's constant, \( N_V \) and \( S \) are the density and spin of vanadium ions, and \( \langle S \rangle \) is the thermal average of \( S \). The \( p-d \) exchange term \( J_{pd} \) is defined by the interaction Hamiltonian as

\[
H = -J_{pd} \sum_i \delta(r - R_i) S_i \cdot s
\]

where \( S_i \) is the vanadium spin at site \( R_i \) and \( s \) is the spin of the carrier at \( r \). The formulation given by Eq. (1) ignores spin-spin correlation of carriers. In Fig. 5 we show the results of the fit to \( \Delta \rho = \rho(B) - \rho(0) \) at several temperatures near and above \( T_C \) for \( \text{Sb}_{1.97}\text{V}_{0.03}\text{Te}_3 \). The room-temperature Hall coefficient was used to determine the carrier density \( p \). \( k_F \) was calculated from \( p \) assuming a spherical Fermi surface.

\[\langle S \rangle \] was found directly from the magnetization data using the equation \( M = N_V g \mu_B \langle S \rangle \), where \( \mu_B \) is the Bohr magneton and \( g = 2 \) is the Lande \( g \) factor. We take \( S = 1 \) based on the analysis of the magnetization data below. Relying on literature values for the upper valence band hole effective mass of \( \text{Sb}_2\text{Te}_3 \) for a single valley \( \left( m^* = 0.08 m_0 \right) \) (Refs. 15 and 16) which we believe to be appropriate at these low temperatures, and making a correction for the multivalley (sixfold degenerate) nature of the band \( \left( m^* = 6^{2/3} m^* = 0.26 \right) \), a value for \( J_{pd} \) of 425 eV Å\(^3\) (or \( N_0 \beta = 5.3 \) eV) is obtained. This \( J_{pd} \) is larger than that for (Ga, Mn) As [3.3 eV (Ref. 14)] when calculated using Eq. (1). A zero field \( \rho_s \) of 0.39 mΩ cm is calculated, which seems to be a factor of 2 larger than one would infer from the experimental data in Fig. 4. In either case, spin-disorder scattering appears to be a significant fraction of the total resistivity.

The peak in \( R_H \) can be understood by considering the anomalous Hall effect,\(^\text{18}\) in which the Hall resistivity \( \rho_H \) is expressed as

\[
\rho_H = R_0 B + R_s M,
\]

where \( R_0 \) is the ordinary Hall coefficient, \( R_s \) the anomalous Hall coefficient, and \( M \) the magnetization of the samples. For temperatures above \( T_C \), \( \rho_H \) is linear in magnetic field up to \( B = 50 \) K with zero intercept implying that the second term in Eq. (3) is insignificant in this temperature range. Below \( T_C \), the transition to the ferromagnetic phase results in a large \( M \) and thus the second term becomes significant. The maximum in \( R_H \) seen in Fig. 3 results from a peak in \( R_s \) that occurs at a temperature close to \( T_C \) in ferromagnetic materials.\(^\text{18}\)

To verify the presence of the magnetic order, we carried out detailed magnetic measurements with the aid of a SQUID magnetometer. Magnetic susceptibility \( \chi \) versus temperature obtained by cooling in a field of 1 K G oriented parallel to the \( c \) axis of the samples is shown in Fig. 6. A value of \( \chi = -3.8 \times 10^{-7} \) emu/g, roughly independent of tempera-
Here, given by Bohr magneton, magnetic susceptibility is well described by a Curie-Weiss law, was found for Sb$_2$Te$_3$ confirming the diamagnetic nature of pure $A^7V^3_7B^6_{13}$ tetradymite semiconductors. The transition to the ferromagnetic state for the samples containing vanadium is very clear as $\chi$ becomes positive and very large below $T_C$. Above the magnetic transition temperature, the magnetic susceptibility is well described by a Curie-Weiss law,

$$\chi = \frac{C}{T - \Theta_p} + \chi_0,$$

(4)

where $C$ is the Curie constant, $\Theta_p$ is the paramagnetic Curie temperature, and $\chi_0$ is the temperature independent diamagnetic lattice term. The fits to the data using Eq. (4) are shown in the inset of Fig. 6 and the fitting parameters are given in Table II. For a given concentration of vanadium in each sample, the magnetic state of the ions can be inferred from the Curie constant using the equation $C = N_V \mu_B^2 \chi_0^2 / 3k_B$. Here, $N_V$ is the concentration of vanadium atoms, $\mu_B$ is the Bohr magneton, $\chi_0$ is the effective Bohr magneton number given by $g(S(S+1))^{1/2}$ with the Landé $g$ factor ($g = 2$) and the spin $S$. We are assuming complete quenching of the orbital angular momentum, which is the usual case for transition-metal ions. We note that the values of $\chi_0$ increase from 1.9 to 2.6 as $x$ increases from 0.01 to 0.03. This might indicate an evolution to a predominantly $3^+$ valence state of vanadium (expected spin only value of $p_{\text{eff}} = 2.83$). We caution, however, that the very low concentrations of vanadium in these samples lead to uncertainty in the determination of $\chi$ and hence $p_{\text{eff}}$. Detailed electron spectroscopy studies would shed more light on this issue.

The magnetization behavior of the structure is shown in Fig. 7. The wide hysteresis loops observed when the field is oriented parallel to the $c$ axis [Fig. 7(a)] are an unambiguous signature of ferromagnetic ordering in these samples. Although not obvious from the plot, the $x = 0.01$ sample shows hysteresis with a small coercive field of approximately 250 G. Such smooth hysteresis loops (no apparent discontinuities) are suggestive of coherent rotation$^{19}$ of spins in this system. Another estimate of the spin of the vanadium ions can be obtained using the equation $M_{\text{sat}} = N_V g \mu_B S$ and the results are given in Table II. Again we see a similar trend in the effective Bohr magneton numbers as the vanadium content increases. When the magnetic field is applied perpendicular to the $c$ axis as shown in Fig. 7(b) practically no hysteresis is detected indicating that the easy axis for magnetization is parallel to the $c$ axis. This may reflect the anisotropic structure of the tetradymite semiconductors.

Perhaps the most spectacular revelation of the long-range magnetic order in the transport data comes from the hysteretic behavior in the transverse magnetoresistance ($B \parallel c$ axis and current $\perp c$ axis) displayed in Fig. 7(c). The large and sharp maxima on the magnetoresistance curve occur at magnetic fields that correspond to zero magnetization (coercive field) and therefore maximum spin disorder in the system. One-half of the width between the maxima matches very well the coercive field values obtained from hysteresis in the magnetization loops. Anisotropy of this system was further probed by investigating the magnetoresistance as a function of the angle between the applied magnetic field and the crystallographic orientation. Figure 8 depicts this data normalized to the $B_{\parallel c} (\theta = 90^\circ)$ value taken at several temperatures for the $x = 0.03$ sample. Below $T_C$, a maximum in $R/R_{B_{\parallel c}}$ develops and grows with decreasing temperature. As temperature decreases, the peak is first symmetric and its position corresponds to the $B \perp c$ orientation, but becomes asymmetric and begins to shift to higher angles below 10 K. The data shown are taken in increasing $\theta$. For data taken in decreasing $\theta$, the peak positions at low temperatures again occur after $B$ crosses the $c$ plane (now at negative $\theta$), i.e., hysteresis is observed.

<table>
<thead>
<tr>
<th>$x$</th>
<th>$C$ [emu K/g]</th>
<th>$\chi_0$ [emu/g]</th>
<th>$\Theta_p$ [K]</th>
<th>$T_c$ [K]</th>
<th>$p_{\text{eff}}^{(1)}$</th>
<th>$p_{\text{eff}}^{(2)}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01</td>
<td>7.610 × 10$^{-6}$</td>
<td>$-4.03 \times 10^{-7}$</td>
<td>12.5</td>
<td>11</td>
<td>1.9</td>
<td>1.5</td>
</tr>
<tr>
<td>0.2</td>
<td>1.705 × 10$^{-5}$</td>
<td>$-4.21 \times 10^{-7}$</td>
<td>21.2</td>
<td>17</td>
<td>2.3</td>
<td>2.1</td>
</tr>
<tr>
<td>0.03</td>
<td>3.598 × 10$^{-5}$</td>
<td>$-4.72 \times 10^{-7}$</td>
<td>23.4</td>
<td>22</td>
<td>2.6</td>
<td>2.3</td>
</tr>
</tbody>
</table>
The temperature dependence of the coercive field $H_c$ for $\text{Sb}_{2-x}\text{V}_x\text{Te}_3$ is shown in Fig. 9. We compare the magnetization values of $H_c$ for $x=0.03$ to the positions of the peaks in the hysteretic pattern of low-temperature magnetoresistance ("+" symbols in Fig. 9). It is found that the hysteretic behavior of both magnetization and transport measurements agree with each other. This correspondence is not surprising since, as the sample becomes demagnetized at $H_c$, the scattering of carriers increases due to spin disorder scattering. This interpretation is also consistent with the angular dependence of the magnetoresistance. The crystal anisotropy may contribute to this effect since the spins clearly only tend to orient themselves along the $c$ axis as evidenced by Fig. 7.

Magnetic-field dependence of the magnetization for $\text{Sb}_{2-x}\text{V}_x\text{Te}_3$ was investigated over a broad temperature region. In the case of a conventional second-order ferromagnetic transition, Arrott plots should give positive slope straight lines given that the magnetic field is high enough so that possible domain or stray field effects are not seen. In these materials, the saturation field at low temperature is near 25 kG as is seen in Fig. 7. Indeed, for fields below 25 kG and especially below the ordering temperature, we see deviation from linear behavior. However, from the high-field extrapolation of the data, we were able to make an estimate of the temperature dependence of the spontaneous magnetization $M_s$ and the Curie temperature $T_C$. This data are shown in Fig. 10. The inset to Fig. 10 indicates that near $T_C$, the curves are parallel and evenly spaced with temperature. From the slight curvature at these fields, one can see that the Curie temperatures estimated here are the lower limits. Arrott plots for $x=0.01$ displayed strong curvature at low temperatures which made it difficult to determine $M_s$ versus $T$ much below $T_C$, thus we only show $x=0.02$ and

![FIG. 7. Field dependence of the magnetization measured at 2 K for $\text{Sb}_{2-x}\text{V}_x\text{Te}_3$ with $x=0.01$ (open circles), $x=0.02$ (filled triangles), and $x=0.03$ (open triangles). In (a), the field is aligned parallel to the $c$ axis. In (b), data for $x=0.03$ is shown for field aligned both parallel and perpendicular to the $c$ axis indicating that the $c$ axis is the easy axis for magnetization. In (c), the magnetoresistance displays hysteresis corresponding to the magnetization data.](image)

![FIG. 8. Transverse magnetoresistance as a function of angle between the applied magnetic field and the $c$ plane of $\text{Sb}_{1.97}\text{V}_{0.03}\text{Te}_3$ at several temperatures. Here $\theta=0^\circ$ corresponds to $B \perp c$.

![FIG. 9. Temperature dependence of the coercive field determined from magnetization hysteresis loops of $\text{Sb}_{2-x}\text{V}_x\text{Te}_3$ (open and closed symbols). "+" symbols designate peak positions in magnetoresistance obtained from hysteresis measurements (see Fig. 7). A direct correspondence between magnetization and magnetoresistance is apparent.](image)
The dominant interaction is antiferromagnetic due to the superexchange among the magnetic ions and free carriers. For Mn-based II-VI DMS's where the transition-metal ion concentration is high and carrier concentration low, the dominant interaction is antiferromagnetic due to the superexchange interaction.\textsuperscript{2} In III-V DMS's, the maximum Mn concentration is lower (up to ~7% using low-temperature MBE techniques); however, in the presence of a high hole density, a carrier mediated ferromagnetic interaction is invoked which has been discussed within a number of frameworks including the Ruderman-Kittel-Kasuya-Yosida (RKKY) interaction\textsuperscript{21} and a mean-field theory.\textsuperscript{25} The Zener model description,\textsuperscript{23} a variant approach, implies that the Curie temperature $T_C$ is determined by a competition between the ferromagnetic and antiferromagnetic interactions, and predicts that $T_C$ increases with hole concentration. In our samples with very dilute content of vanadium and large carrier (hole) background, a likely scenario for the onset of the ferromagnetic state is the RKKY interaction. However, we cannot rule out alternative theories which challenge the free-carrier based explanation of the ferromagnetism.\textsuperscript{24–26} We wish to note that the less than half filled $d$ shell reflecting the magnetic state of vanadium is unusual in the context of diluted magnetic semiconductors. It has been proposed\textsuperscript{27} that under these conditions the superexchange among the magnetic ions is ferromagnetic rather than antiferromagnetic. The fact that the magnetoresistance can be described well within the Kasyuta theory lends support to the RKKY interaction mediated by holes for the origin of the ferromagnetism, though further study is needed to elucidate the mechanism.

### IV. SUMMARY

In summary, a diluted magnetic semiconductor based on the layered, narrow-band-gap Sb$_2$Te$_3$ doped with vanadium was discovered. Magnetization studies reveal that the easy axis for magnetization lies along the $c$ axis. The sample with composition Sb$_{1.97}$V$_{0.03}$Te$_3$ has a hole concentration of $9.8 \times 10^{19}$ cm$^{-3}$ and a Curie temperature of at least 22 K. Through an effort to incorporate more vanadium ions into the Sb$_2$Te$_3$ structure and the use of known effective acceptors,\textsuperscript{28} there are excellent prospects for achieving higher Curie temperatures via increased densities of both holes and magnetic ions. Existing methods [including MBE (Refs. 29 and 30)] for thin-film growth of Sb$_2$Te$_3$ and related tetradymite structures may help to explore the upper limit of magnetic ion incorporation. These tetradymite-based materials are not in the mainstream of present-day semiconductor technology and interfacing these materials with current thin-film device applications poses a challenge. Nevertheless, one ought to investigate the full scope of the physical properties and new science that the structure might support. We stress that in our system, we move away from the usual environment of a zinc-blende crystal structure and Mn ions that underpin the magnetic properties of the existing DMS's. Therefore these results pave the way for a new parameter space for search and exploration of other diluted magnetic semiconductors based on the $A^2B_3^3$ tetradymite system doped with 3$d$ transition elements.

### ACKNOWLEDGMENTS

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