Magnetic two-dimensional electron gases with high Curie temperatures at $LaAlO_3/SrTiO_3$:Fe interfaces

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Spin-polarized two-dimensional electron gas (2DEG) at the interface of two insulating perovskite oxides has been a focus of intensive studies in recent years. So far all attempts to construct magnetic 2DEG are based on the selection of an appropriate buffer layer or cap layer in $SrTiO_3$-based heterostructures, and the magnetic effect thus produced on 2DEG is indirect and weak. Here, we fabricated the 2DEG based on Fe-doped $SrTiO_3$ that is superparamagnetic rather than diamagnetic like $SrTiO_3$. In addition to good metallicity, considerable Kondo effect, and negative magnetoresistance, the most striking observation of the present work is the occurrence of the anomalous Hall effect up to room temperature. This is transport evidence for the existence of spin-polarized 2DEG at high temperatures. As suggested by the monotonic increase of Curie temperature with carrier density, the magnetic exchange between magnetic ions could be mediated by the itinerant electrons of the 2DEG. The present work opens an avenue for the exploration of spin-polarized 2DEG.

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Two-dimensional electron gas (2DEG) at the interface between two band insulators $LaAlO_3$ (LAO) and $SrTiO_3$ (STO) has been a focus of intensive studies in recent years due to its exotic properties such as coexisting 2D superconductivity and 2D magnetism, strong gate effect, and efficient charge-to-spin conversion [1–5]. Among them, the 2D magnetism is of special interest because in a sense it suggests the possibility to gain spin-polarized 2DEG that is centrally important for spintronics. In the past decade, different techniques including cantilever magnetometer [5], scanning superconducting quantum interference device (SQUID) magnetometer [1,6,7], magnetic force microscopy [8], and x-ray magnetic circular dichroism [9,10] have been employed for a direct magnetic measurement of the LAO/STO interface. Transport investigations such as magnetoresistance (MR) [11,12], Kondo effect [11], anisotropic MR [13], and Hall resistance [14–17] were also conducted. However, a lot of issues on the magnetism of the LAO/STO interface remain to be addressed.

In general, the magnetic order of the 2DEG, when established, is usually weak and appears at very low temperatures, which has strongly impeded both the fundamental and applied researches. Since the first discovery of the 2D magnetism of the 2DEG in 2007 [11], various attempts have been devoted to the fabrication of highly spin-polarized 2DEG at as high as possible temperatures. Different from other physical quantities, anomalous Hall effect (AHE) is an irrefutable fingerprint of ferromagnetic (FM) order for conducting materials. The earliest report on AHE was given by Joshua *et al.* [17] for the LAO/STO 2DEG at 2 K. Subsequently Gunkel *et al.* [15] found the AHE at the conducting $NdGaO_3$/STO interfaces in the temperature range below 10 K. To enhance the AHE, people have tried to fabricate slightly complex heterostructures by inserting an ultrathin magnetic layer between LAO and STO, such as the LAO/$EuTiO_3$/STO structure by Stornaiuolo *et al.* [14] and the LAO/$La_{7/8}Sr_{1/8}MnO_3$/STO structure by Zhang *et al.* [18]. In this manner, the Curie temperature was promoted to ~10 or ~30 K. As an alternative, Moetakef *et al.* [12] designed a 2DEG by sandwiching an ultrathin STO layer between two magnetic $GdTiO_3$ films. In this case, hysteretic MR is observed up to 10 K although no AHE was reported [12], which implies that the STO is indeed magnetized. Despite these progresses, there is still a long distance to go for gaining well-spin-polarized 2DEGs. Particularly, it remains challenging to get magnetic 2DEGs with high Curie temperatures.

So far, all attempts to fabricate magnetic 2DEG have started from the STO-based heterostructures, via selecting appropriate buffer layers or cap layers. Since the 2DEG resides in diamagnetic STO, however, the effects thus produced are indirect and usually very weak. Notably, STO can be made magnetic when partially replacing Ti^{3+} ions with Fe^{3+} ions. This raises an interesting question on what will happen when the 2DEG resides in magnetic STO (STO:Fe) and travels among magnetic ions. Here, we report the formation of a

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high Curie temperature magnetic 2DEG at the LAO/STO:Fe interface. In addition to good metallicity, considerable Kondo effect, and negative MR, the most remarkable observation is the occurrence of the AHE up to room temperature. This is transport evidence for the existence of magnetic order in 2DEG at high temperatures.

Three LAO/STO:Fe interfaces have been fabricated by growing LAO films on TiO_2-terminated (001)-$SrTiO_3$ single-crystal substrates doped by 0.05 wt. % Fe using the technique of pulsed-laser deposition (excimer laser with a wavelength of 248 nm). Here the content of Fe in STO was chosen so that it introduces sufficient magnetism but does not considerably deteriorate the mobility of the 2DEG. During the deposition, the substrate temperature was kept at 750 °C and the oxygen pressures (P_{O2}s) were fixed to 10^{-4}, 10^{-5}, or 10^{-6} mbar, respectively. The laser fluence was 1.5 J cm^{-2} and the repetition rate was 1 Hz. After growth, the samples were naturally cooled to room temperature without changing oxygen pressure. The LAO layer was 5 unit cells (u.c.) in thickness, as indicated by reflection high-energy electron diffraction (RHEED).

Surface morphology of the samples was measured by atomic force microscope (SPI 3800N, Seiko). Ultrasonic wire bonding (Al wires of 20 μm in diameter) was used for electric contacts. The van der Pauw geometry was adopted for resistive measurements. Both longitudinal and transverse resistances were measured, using the Quantum Design physical property measurement system with an applied current of 10 μA. For the investigation of the gating effect, a gate voltage was applied to a copper back gate while the 2DEG was grounded. The leakage current was lower than 10 nA. The magnetic measurements were carried out by a vibrating-sample magnetometer SQUID.

Figure 1(a) shows the oscillation of the RHEED intensity, collected in the growth process of a typical LAO cap layer. It reveals a layer-by-layer epitaxial growth of LAO on STO:Fe. The time required by each LAO layer is about 60 s. Figures 1(b) and 1(c) are the RHEED patterns before and after the growth of the LAO layer, respectively. Sharp RHEED peaks can be clearly seen, indicating the high crystal quality of the substrate and the LAO film. Figure 1(d) is the typical morphology of the sample grown under a P_{O2} of 10^{-4} mbar. The film surface is atomically flat with regular steps of the height of 1 u.c.

All three LAO/STO:Fe interfaces exhibit the typical feature of 2D conduction as confirmed by anisotropic MR [19], and thus host 2DEGs. Figure 2(a) shows the temperature (T) dependence of the sheet resistance (R_S) of LAO/STO:Fe. Two features can be identified from the R_S -T curves. Firstly, all 2DEGs are well metallic; the sheet resistance decreases by one order of magnitude when cooled from 300 down to 30 K. Secondly, a minor but visible resistance upturn appears below 30 K, due to Kondo scattering [19]. Decrease in P_{O2} affects mainly the high-temperature resistance, resulting in an obvious reduction in R_S. These phenomena seem to be similar to those observed in the 2DEG at the LAO/STO interface [11]. However, a close inspection indicates that the low-temperature sheet resistance of the present 2DEG is considerably large. It is ∼1.8 kΩ/□ at 2 K in contrast to 0.3 kΩ/□ for the STO-based 2DEG fabricated with the same condition (10^{-4} mbar). Obviously, Fe^{3+} ions in STO produce additional scattering to itinerant electrons.

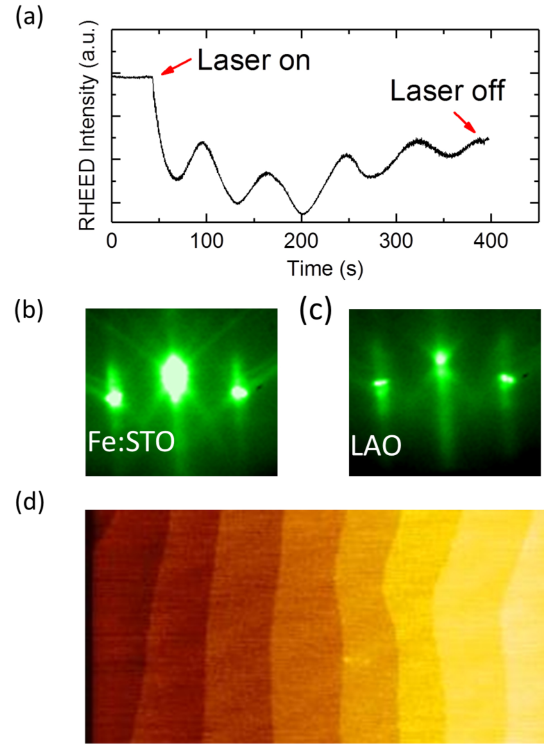

FIG. 1. (a) Typical RHEED spectrum showing an intensity oscillation corresponding to the growth of LAO on STO:Fe. (b), (c) RHEED patterns before and after the deposition of the LAO cap layer, respectively. (d) Morphology of the LAO layer on STO:Fe ($P_{O2} = 10^{-4}$ mbar), recorded by atomic force microscope. Image size is $5 \times 2.5\ \mu\text{m}^2$.

Corresponding to the P_{O2}s of 10^{-6}, 10^{-5}, and 10^{-4} mbar, the carrier density (n_S) is 1.1×10^{14}, 9.1×10^{13}, and $5.8 \times 10^{13}\ \text{cm}^{-2}$ at 300 K and 5.9×10^{13}, 5.0×10^{13}, and $3.7 \times 10^{13}\ \text{cm}^{-2}$ at 2 K. It nearly linearly decreases as temperature varies from 300 to 2 K, i.e., the charge carriers are continuously frozen out with the decrease of temperature. This is different from the STO-based 2DEG, for which the carrier density decreases upon cooling at relatively low speeds. Therefore, additional scattering from magnetic ions has resulted in considerable charge localization.

Iron doping also influences Hall mobility (μ_H). Although μ_H exhibits the same temperature dependence as that of the 2DEG at the LAO/STO interface, i.e., it is low at room temperature and increases rapidly upon cooling, its value is considerably low. Take the 2DEG fabricated under a P_{O2} of 10^{-4} mbar as an example. The Hall mobility is on the order of $\sim 6\ \text{cm}^2/\text{Vs}$ at room temperature, comparable to that of the STO-based 2DEG, and $110\ \text{cm}^2/\text{Vs}$ at 30 K, much lower than $10^3\ \text{cm}^2/\text{Vs}$ for the LAO/STO 2DEG. When cooled below 30 K, the μ_H-T relation exhibits a visible downward curvature due to Kondo scattering.

An issue of special interest is the possible magnetic effect produced by Fe dopants. This effect, if it exists, should have a reflection in Hall resistance (R_{xy}). As an example, in Fig. 3(a) we show the magnetic field (B) dependence of the Hall resistance of the 2DEG formed under a P_{O2} of 10^{-6} mbar. At first glance, the R_{xy}-B relation is well linear, indicating the

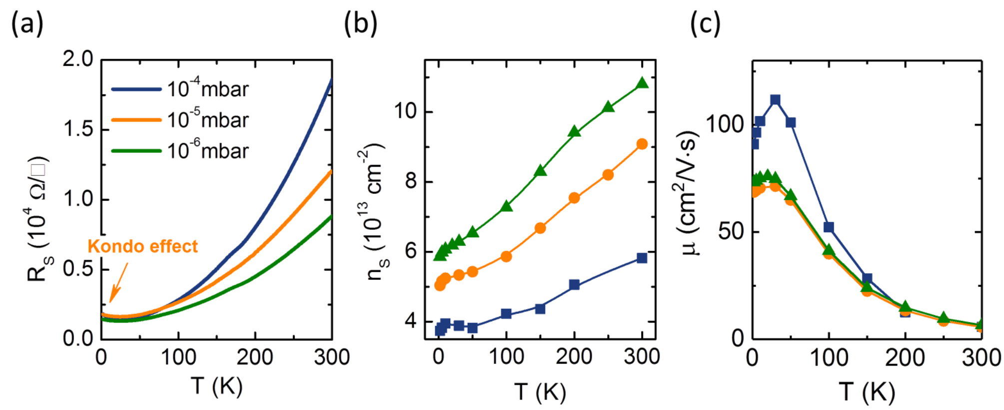

FIG. 2. (a) Temperature dependence of sheet resistance for the LAO/STO:Fe interfaces fabricated under different oxygen pressures. (b), (c) Corresponding sheet carrier density and Hall mobility, respectively.

dominative role of the normal Hall effect. The R_{xy}-B slope varies from −10.7 to −5.8 Ω/T as temperature grows from 2 to 300 K, in the expected range for the 2DEG. However, the derivative of R_{xy} with respect to B shows visible anomalies [Fig. 3(b)], forming an inverse peak around $B = 0$. It means the occurrence of a stepwise transition for Hall resistance as the magnetic field sweeps through the origin, i.e., the appearance of the AHE. The peak height is small at 2 K and grows gradually as temperature sweeps from 2 to 30 K, and then reduces upon further heating. Strikingly, this anomaly remains sizable up to 300 K. This is evidence from transport measurements for the presence of magnetic order in 2DEG at so high a temperature.

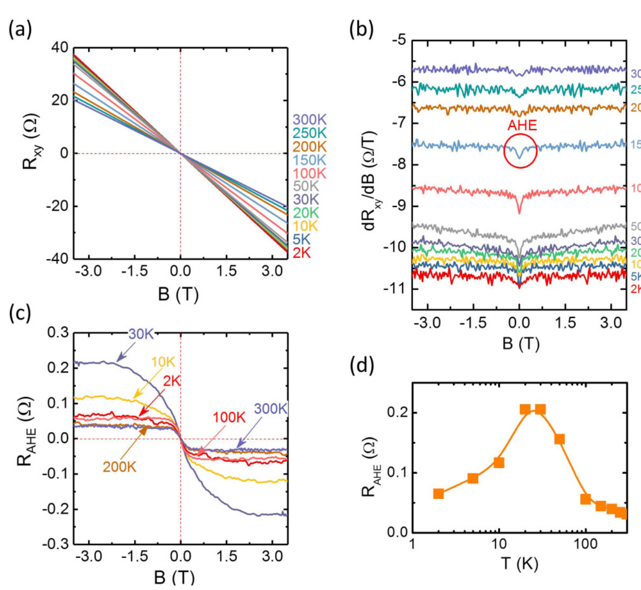

FIG. 3. (a) Hall resistance as a function of perpendicular magnetic field for the 2DEG fabricated under the P_{O2} of 10^{-6} mbar. (b) Derivative of Hall resistance with respect to magnetic field. Inverse steep peaks marked by red circle indicate the occurrence of AHE. (c) Anomalous Hall resistance deduced by subtracting the linear part from the R_{xy}-B curves in (a). Here, the curves in (b) and (c) have been symmetrized. (d) Saturation anomalous Hall resistance as a function of temperature. The decrease of R_{AHE} below 30 K is ascribed to the appearance of Kondo effect.

After subtracting the linear background and the superparamagnetic effect of STO:Fe [19] from the R_{xy}-B curve, anomalous Hall resistance (R_{AHE}) can be determined. Figure 3(c) shows the deduced R_{AHE}-B dependence obtained at different temperatures. All R_{AHE}-B curves are step-shaped, with the most dramatic changes taking place between −1.5 and 1.5 T. The saturation R_{AHE} is very small on average. It is ∼0.06 Ω at 2 K, and rapidly grows to a maximal value of ∼0.19 Ω at 30 K, then decreases slowly with the further increase of temperature [Fig. 3(d)]. However, the AHE remains visible up to the temperature of 300 K. This is in sharp contrast to the previously reported results [15], which show that the AHE-like behavior survives only below 10 K for the 2DEG confined to the LAO/$EuTiO_3$/STO or $NdGaO_3$/STO interface. Obviously, the incorporation of Fe^{3+} produces a more robust exchange coupling among magnetic ions. The AHE is similar for the other two samples, although the Curie temperature is slightly low, ∼50 K when $P_{O2} = 10^{-4}$ mbar and 150 K when $P_{O2} = 10^{-5}$ mbar [19]. It is therefore a general feature of the 2DEG at the LAO/STO:Fe interface. Although there are reports on oxygen vacancy-induced interfacial magnetism for LAO/STO [10], we did not observe AHE in the LAO/STO interfaces prepared under the oxygen pressures adopted here. Therefore, Fe dopants in STO play a crucial role in inducing the AHE.

The decrease in Curie temperature for the samples from $P_{O2} = 10^{-6}$ mbar to $P_{O2} = 10^{-4}$ mbar can be ascribed to the decrease in carrier density. On the analogy of diluted magnetic semiconductor, the magnetic interaction between magnetic ions grows with the density of charge carriers [20]. In fact, we also tried to tune the carrier density of our 2DEGs by gating effect and observed an enhancement of the AHE when positively gated and a weakening when negatively gated [19]. This result reveals the important role of carrier density for magnetic ordering.

As well established, R_{AHE} is proportional to the product of R_S^2 and magnetization (M) [21]. The magnitude of R_{AHE} is small, indicating that the magnetization of our LAO/STO:Fe interface is low. This is understandable since the content of Fe ions in STO:Fe is very low, only ∼0.05 wt. %. Provided that the magnetic signals exclusively come from Fe^{3+} ions and each Fe^{3+} ion contributes $2\,\mu_B$, the expected magnetization will be $\sim 0.14\,\mathrm{emu/cm^3}$. Although the magnetic contributions

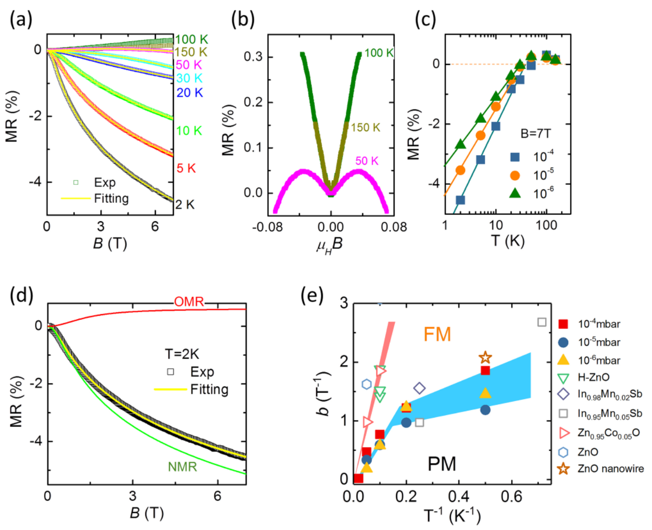

FIG. 4. (a) Magnetoresistance as a function of perpendicular magnetic field for the LAO/STO:Fe interface prepared under the oxygen pressure of 10^{-4} mbar. Experimental and curve-fitting results are presented by symbols and solid lines, respectively. Here, the curves under positive and negative magnetic field have been symmetrized. For clarity, only the positive part is shown. (b) Magnetoresistance as a function of the product of Hall mobility and magnetic field, shown for selected temperatures where the ordinary MR effect dominates. Here, the MR-$\mu_H B$ curves have been symmetrized for clarity. (c) A summary of the magnetoresistance recorded at 7 T for all three samples. (d) A demonstration of data fitting at 2 K, where two components (OMR and NMR) are all presented. (e) Fitting parameter b as a function of reciprocal temperature. The cyan and pink shaded areas mark the distribution of the present experimental data and reported data for diluted magnetic semiconductors [27–31], respectively.

from Ti^{3+} ions cannot be completely ruled out, the total magnetization could still be fairly low. We have performed a magnetic measurement for the STO:Fe substrate and found a saturation magnetization of $\sim$0.26 emu/cm^3 [19]. Therefore, our 2DEG could be a diluted magnetic 2DEG with a weak AHE.

The variation of R_{AHE} with temperature is complex, rather than the expected monotonic growth upon cooling. Notably, R_{AHE} turns from increase to decrease when the Kondo effect sets in. This implies a close relation between these two processes [22]. Possibly, the magnetic order has been affected by Kondo screening below 30 K, which results in the decrease of R_{AHE}. According to the experimental results, carrier density exhibits a rapid decrease upon cooling. Take the sample prepared at $P_{O2} = 10^{-6}$ mbar as an example. From 300 to 2 K, the carrier density is nearly halved. Consequently, the magnetic coupling could be weakened. It means that, at low temperatures, the effect of Kondo scattering may be able to compete with that of magnetic ordering. Since the Kondo screening is incomplete even at 2 K [19], R_{AHE} remains sizable although it is reduced.

In addition to the Hall effect, MR also contains important information on the magnetic state of the 2DEG. As an example, in Fig. 4(a) we show the MR of the 2DEG ($P_{O2} = 10^{-4}$ mbar), measured with perpendicular fields. The MR, defined as $[\rho(B)-\rho(0)]/\rho(0)$, is negative at low temperatures. Although it is not large in magnitude (4.6% at 2 K and 7 T), its dependence on applied field is distinct: At low temperatures the MR-B curve forms a sharp maximum around $B = 0$, rather than a broad one like the 2DEG at the LAO/STO interface [23,24]. With the increase of temperature, the magnitude of the MR decreases gradually, and undergoes a negative to positive crossover at 50 K, which leads to a slightly complex MR-B dependence [Fig. 4(b)]. Above 100 K, positive MR obeying Kohler's rule [Fig. 4(b)] prevails. It is an ordinary MR (OMR) that arises from the magnetic-field-induced orbital effect [23]. The MR-B curves in other two samples are similar to the present sample except for slight differences in detailed shapes [19]. Figure 4(c) exemplifies the temperature dependence of the MR recorder at 7 T for all three samples. Negative MR (NMR) emerges at very low temperature, rapidly decreases in magnitude as temperature increases, and turns to OMR above $\sim$50 K.

The NMR deserves special attention. It is also observed in the 2DEG at the LAO/STO interface and usually attributed to the depression of weak localization by perpendicular magnetic field [24,25]. For the present 2DEG, however, it may have a different origin, i.e., the field-orientated alignment of localized moments. In this case, MR will vary with applied field obeying an equation obtained by Khosla and Fischer [26] based on the hypothesis of MR $\propto M^2$,

$$MR = -a^2 \ln(1 + b^2H^2) + \frac{c^2H^2}{1 + d^2H^2}, \tag{1}$$

$$a^2 = A_1 J D(\varepsilon_F)[S(S+1) + \langle M^2\rangle], \tag{2}$$

$$b^2 = \left[1 + 4S^2\pi^2\left(\frac{2J D(\varepsilon_F)}{g}\right)^4\right]\frac{g^2{\mu_B}^2}{(\alpha k_B T)^2}. \tag{3}$$

The first term of Eq. (1) describes the NMR, and the second term is the OMR when two species of charge carriers coexist. In Eq. (2), A_1 is a measure of the spin-scattering contribution to MR, J is the exchange interaction energy, $D(\varepsilon_F)$ is the density of states at the Fermi level, and $\langle M^2\rangle$ is the average of the square of the magnetization. In Eq. (3), μ_B is the Bohr magneton, k_B is the Boltzmann constant, α is a numerical factor that is on the order of unity, S and g are the total spin and the Landé g factor of the localized magnetic moments, respectively.

As shown by the solid curves in Fig. 4(a), Eq. (1) well reproduces the experimental results in all measured temperature range adopting the fitting parameters tabulated in Supplemental Material [19]. Moreover, the fitting parameter b is proportional to $1/T$ in the high-temperature range, in good agreement with Eq. (2). The deduced b-$1/T$ slope is $\sim$7.4 K/T^2. These behaviors are very similar to those of diluted magnetic semiconductors which exhibit a b-$1/T$ slope of 18.5 K/T^2. We also tried to fit the MR-B relation to the model of weak localization, and the results are not satisfactory [19]. These results strongly suggest that the NMR stems from a coherent magnetic scattering of the itinerant electrons by correlated localized moments, supporting the conclusion of the Hall effect.

Different from the AHE that persists to room temperature, the NMR is invisible above 100 K. This may be ascribed to the different magnetization dependence of these two physical

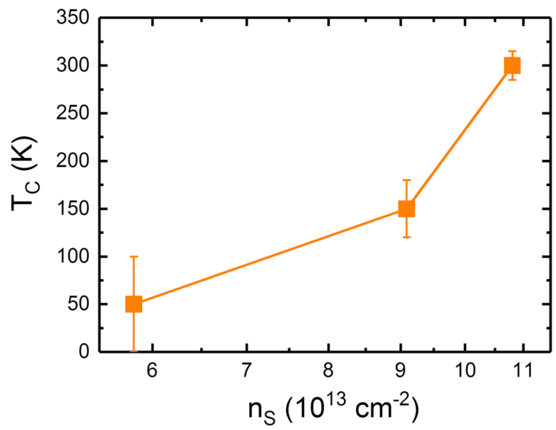

FIG. 5. Curie temperature of the 2DEG as a function of carrier density determined at room temperature.

quantities. As well established, R_{AHE} is proportional to the product of R_S^2 and M while NMR to M^2. At high temperatures, M may be very small and its effect on NMR has been overwhelmed by OMR. However, R_{AHE} could be finite since the decrease in M can be compensated by the increase in sheet resistance.

The origin of the FM order in the present 2DEG is a further issue to be addressed. We studied the magnetic property of STO:Fe and found the M-B relations well described by the Langevin function with a magnetic moment of 6.86 μ_B [19]. It means that the STO:Fe is superparamagnetic. However, the unexpectedly large magnetic moment implies that the magnetic ion tends to magnetize its surroundings. This tendency could be further enhanced in the interfacial layer since the 2DEG there will mediate a magnetic exchange between localized moments, playing the role of itinerant electrons in diluted magnetic semiconductors. In this scenario, the magnetic ordering of the 2DEG will be closely related to carrier density. Indeed, we found a monotonic increase of the Curie temperature of our 2DEG with carrier density (Fig. 5). Although the performance of our 2DEG still has room for further improvement, the present work demonstrates the great potential of magnetic substrate in the fabrication of spin-polarized 2DEG.

In conclusion, magnetic 2DEGs have been successfully fabricated based on superparamagnetic Fe-doped STO. In addition to good metallicity, considerable Kondo effect, and obvious negative magnetoresistance, the most remarkable discovery is the occurrence of anomalous Hall effect up to room temperature. The Curie temperature exhibits a monotonic increase with carrier density, indicating the importance of the itinerant electrons of the 2DEG for the establishment of the magnetic order. The present work reveals the irreplaceable role of the magnetic substrate where the 2DEG resides in the fabrication of spin-polarized 2DEG.

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