Quantum longitudinal and Hall transport at the $\text{LaAlO}_3/\text{SrTiO}_3$ interface at low electron densities

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We examined the magneto-transport behavior of electrons confined at the conducting $\text{LaAlO}_3/\text{SrTiO}_3$ interface in the low sheet carrier density regime. We observed well-resolved Shubnikov-de Haas quantum oscillations in the longitudinal resistance, and plateau-like structures in the Hall conductivity. The Landau indices of the plateaus in the Hall conductivity data show spacing close to 4, in units of the quantum of conductance. These experimental features can be explained by a magnetic breakdown transition, which quantitatively explains the area, structure, and degeneracy of the measured Fermi surface.

Keywords:

- A. Quantum oscillations
- B. Two-dimensional electron gas
- C. Magnetic breakdown
- D. Strontium titanate

1. Introduction

Recently, there has been significant interest in quantum transport [1–7] and the electronic structure [8–14] of quasi-two-dimensional electron gases (q2DEGs) generated in SrTiO_3 , in which a variety of fascinating properties have been observed [15,16]. However, there are currently few measurements which clearly resolve the nature of the electronic band structure of these q2DEGs. Although Shubnikov-de Haas (SdH) quantum oscillations, which directly measure the Fermi surface, have been observed [1–7,17], they were often poorly resolved. In particular, the Fermi surface constructed from these measurements typically only accounts for a small fraction of the carriers measured by the Hall effect.

Compared with many conventional semiconductors, SrTiO_3 has carriers of much larger effective mass, m^* , which results in a significantly smaller separation of Landau levels in the presence of a magnetic flux B , $heB/2\pi m^*$ (h is the Planck constant and e is the electronic charge). Therefore, it is correspondingly more challenging to realize truly 2D quantum transport in SrTiO_3 . A variety of techniques have been utilized to approach the 2D limit in SrTiO_3 , including delta-doping [1,4,7], electrostatic field-effect doping [18–22] and heterostructuring [6,15,16,23]. In the latter category the q2DEG formed between TiO_2 -terminated {100} SrTiO_3 and LaAlO_3 is intensively studied [15,16]. It has been observed that $\text{LaAlO}_3/\text{SrTiO}_3$ heterostructures grown at lower temperature give relatively lower sheet carrier density and higher mobility [2]. A continuous increase in mobility with decreasing carrier density have been demonstrated when $\text{LaAlO}_3/\text{SrTiO}_3$ heterostructures were tuned by LaAlO_3 surface treatments such as charges and adsorbates [24]. These studies suggest promising avenues to explore quantum transport in the

low electron density regime, possessing simultaneously favorably high mobility and reduced subband complexity.

In this work, we studied the magneto-transport behavior of the q2DEG confined at the $\text{LaAlO}_3/\text{SrTiO}_3$ interface in the low sheet carrier density regime. Our results reveal two compelling features: 1) the presence of plateaus in the Hall conductivity that can be assigned to Landau filling indices with an interval close to 4, implying a 4-fold degeneracy in the band structure; 2) a transition of the frequency of the SdH quantum oscillations (from f to $\sim 3f$) with increasing magnetic field. These features can be understood by considering magnetic breakdown orbits and quantitatively account for all of the carriers.

2. Materials and methods

We use $\text{LaAlO}_3/\text{SrTiO}_3$ heterostructures of low sheet carrier density and high mobility [2,3,5]. The samples were prepared by depositing LaAlO_3 thin films on TiO_2 -terminated {100} SrTiO_3 substrates by pulsed laser deposition, at 650 °C, under 1.3×10^{-3} Pa of O_2 [2]. After growth, the samples were in-situ post-annealed at 550°C, in 2×10^{-4} Pa of O_2 for 1 hour to remove macroscopic amounts of oxygen vacancies. The samples were further treated by writing surface charge using a biased conductive atomic force microscope (CAFM) probe [24] or using a capping layer [5] to produce Hall mobilities up to $10,000 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ (for more details, see discussion in Supplementary Material, Section A). Here we present transport measurements of four different samples. The details of the heterostructures and surface processing are summarized in Table 1. The samples B & C were treated by CAFM under a bias of -8 V, at ambient environment with a relative humidity of $\sim 35\%$. The sample D was capped with a 140 nm amorphous LaAlO_3 (a-LAO) layer deposited at room temperature. All samples were measured

using a standard Hall bar configuration with a current in a range of 0.2-1 μA . The electrical contacts to the buried q2DEG were made by ultrasonic bonding with Al wires. The samples A, C & D were measured in a ^4He cryostat with a ^3He insert. The sample B was measured in a dilution refrigerator with a base temperature of 10 mK. All of these samples showed metallic conduction down to low temperatures (see Supplementary Material, Section B).

3. Results

In Fig. 1 we show the raw longitudinal and Hall resistances, R_{xx} and R_{xy} , as a function of B for the four samples, measured at $T \leq 0.5$ K. Significant SdH oscillations are observed in all cases. In the samples A & C [Figs. 1(a) & 1(c)] the amplitude of oscillations in R_{xx} at $B > 9$ T is more than 50 % of the total resistance, indicating the high quality of the samples. $R_{xy}(B)$ is close to linear except for the superimposed plateau-like structure (the slight non-linearity for sample B may originate from parallel conduction channels). From the low-field ($B < 5$ T) Hall coefficient R_H , the sheet carrier density $n_{\text{Hall}} = -1/eR_H$ and Hall mobility $\mu = R_H/R_{xx}(0)$ were evaluated to be in the range of 4.7 to $7.5 \times 10^{12} \text{ cm}^{-2}$ and $5,500$ to $10,000 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$, respectively [Table 1].

As shown in Fig. 1, the plateau-like features in $R_{xy}(B)$ become more significant with increasing B . In the inset of each panel we plot the Hall conductivity, $G_{xy} = R_{xy}/(R_{xx}^2 + R_{xy}^2)$, versus B in the high field range. At first glance these plateaus in G_{xy} can be approximately assigned to the quantum filling indices with an interval close to 4. To better understand the indices of these plateaus, in Fig. 2 we plot the high field $G_{xy}(B)$ and its derivative in the same figure for each sample, and examine the corresponding values of G_{xy} at the local maxima of dG_{xy}/dB . We found that the plateau positions are not precisely at integer multiples of e^2/h and the indexing shows both odd and even values in different samples, suggesting that we are not in a purely single-band

quantum Hall regime. These observations might be related to the fact that more than one effective mass carrier is involved in the quantization, as discussed below. Nonetheless the $\sim 4e^2/h$ spacing of G_{xy} implies an apparent degeneracy in the electronic structure of the q2DEG.

Next we analyze the quantum oscillations in $R_{xx}(B)$ and $R_{xy}(B)$ in more detail. In the following we focus on the data from the sample A, but similar results are found for the other samples. To clearly resolve the oscillations from the non-oscillatory background we performed second and first order differentiation of $R_{xx}(B)$ and $R_{xy}(B)$ respectively, using the data shown in Fig. 1(a) (see also Supplementary Material, Section B). Figure 3(a) shows dR_{xx}^2/dB^2 and dR_{xy}/dB versus $1/B$. Two distinct sets of oscillations in dR_{xx}^2/dB^2 are found, with a crossover in behavior around ~ 4.3 T ($1/B = 0.23 \text{ T}^{-1}$). A Fourier transform (FT) of these dR_{xx}^2/dB^2 data gives two frequencies, $f_{xx1} \sim 20$ T and $f_{xx2} \sim 60$ T [Fig. 3(b)]. The inverse FT shows that the oscillations in low field range ($B < 4.3$ T) correspond to f_{xx1} and the oscillations in high field range ($B > 4.3$ T) correspond to the superposition of f_{xx1} and the dominant f_{xx2} (see Supplementary Material, Section C).

Further insight is obtained by performing a standard fan diagram analysis of the oscillations, where we plot the index of the dR_{xx}^2/dB^2 extrema versus $1/B$. As shown in Fig. 3(c), the magnitudes of the slope of the fan diagram in the low and high field regimes, S_L and S_H respectively, agree well with f_{xx1} and f_{xx2} . The crossover of S_L and S_H corresponds to the transition of the two sets of oscillations. Here in the high field range of the fan diagram analysis only the oscillations which correspond to f_{xx2} have been distinguished. Analysis of the oscillations in dR_{xy}/dB gives similar results [Figs. 3(a), 3(b) & 3(c)], although the absolute values of the frequencies and slopes are slightly smaller. This difference is ascribed to the relatively lower resolution of the oscillations in dR_{xy}/dB compared to those in dR_{xx}^2/dB^2 .

From the temperature dependence of the amplitude of the oscillations in R_{xx} we obtain a cyclotron effective mass $m^* = 0.9 \pm 0.1 m_e$ at $B \approx 4$ T and $m^* = 1.30 \pm 0.04 m_e$ at $B \approx 11$ T [Table 1 & Supplementary Material, Section D)]. The estimated Dingle temperature, T_D , is ~ 1.3 (~ 2.0) K in the high- (low-) field range, smaller than previous reports in similar q2DEGs [1,2], indicating relatively less broadening of the Landau levels in the present samples.

4. Discussion

We now discuss the possible electronic structures which can explain the ~ 4 -fold degeneracy, the S_H/S_L ratio, and the effective mass variation. First of all, we can exclude Zeeman spin splitting as the source of the field-dependent frequency change of the SdH oscillations, since it would give $S_H/S_L = 2$, rather than 3. The conduction band of bulk SrTiO₃ consists of a light band and a heavy band which is relatively close in energy, as well as a split-off band that is expected to exceed the Fermi level in these samples of low carrier density [9,25–28]. Quantum confinement at the interface produces a series of 2D subbands [9,11,12] derived from the light and heavy bands, forming the q2DEG. We consider a case where one light and one heavy subband are occupied, as suggested theoretically for a q2DEG of comparable carrier density with our present samples [9]. Figure 4(a) shows the schematic Fermi surface, in which the inner circle and the outer star represent the light and the heavy subbands respectively. In this scenario it is natural to assign S_L and m^* (low- B) to the circle and S_H and m^* (high- B) to the one of two possibilities: the star or magnetic breakdown (MB) orbits between the star and the circle [26]. The former can be ruled out since it predicts no degeneracy.

In Fig. 4(a) we sketched two representative MB orbits, MB1 (black and shaded) and MB2 (yellow dashed). We note that MB1 is the lowest order closed breakdown orbit (2nd) with

smallest area and effective mass, and would be the first expected to be observed. Although it is possible to construct other MB orbits, they are not responsible for the present observations, being ruled out by a combination of their inappropriate degeneracy, m^* , or enclosed area, following application of the same discussions as below. We assume a Landé g -factor of ~ 2 , as has been observed in bulk SrTiO_3 [29], and in weak-localization studies of the $\text{LaAlO}_3/\text{SrTiO}_3$ interface [13]. In this case, using $m^* = 1.3 m_e$, the Zeeman spin splitting ($g\mu_B B$, where μ_B is the Bohr magneton) is comparable with the Landau splitting ($heB/2\pi m^*$): 1.3 meV vs 0.9 meV at $B = 10$ T. Hence, the intrinsic spin degeneracy of 2 should be lifted at all fields [7]. However, the spin-split up (down) Landau levels may overlap with the adjacent down (up) levels when the splitting energies of the two effects are close to one another, causing apparent spin degeneracy that cannot be resolved due to the finite energy resolution of the SdH oscillation measurements (see discussion in Supplementary Material, Section E). Hence the apparent 4-fold degeneracy observed in $G_{xy}(B)$ can be explained by either, a) the 4 equivalent MB1 orbits without spin degeneracy or b) the 2 equivalent MB2 orbits with effective spin degeneracy.

From the Onsager relation the frequency of the SdH oscillations (or the slope in the fan diagram) is proportional to the enclosed area of the electronic orbit at the Fermi surface, A . The relation between the two dimensional carrier density, n_{2D} , and A is, $A = 4\pi^2 \times n_{2D} / \text{degeneracy}$. With the light electron density $n_l = eS_L/h$ (no spin degeneracy case) and heavy electron density $n_h = n_{\text{Hall}} - n_l$, we calculated the A of light and heavy subbands, and thus the A of MB orbit, based on the geometrical relation shown in Fig. 4 (a). We found that in the case a) $A(\text{MB1})$ is $\sim 3A(\text{circle})$, while in the case b), with presumed 2-fold spin degeneracy, $A_F(\text{MB2})$ is $\sim 2.4A(\text{circle})$. Thus the case a) matches the observed S_H/S_L (or f_{xx2}/f_{xx1}) ratio of 3. In addition, from the definition of the

cyclotron effective mass [30,31], $m^* = \frac{\hbar^2}{8\pi^3} \frac{\partial A}{\partial E} \big|_{E=E_F}$, and the geometrical relations shown in Fig.

4(a), we have $m^*(\text{MB1}) \cong \frac{1}{4}m_h^* + \frac{3}{4}m_l^*$ and $m^*(\text{MB2}) \cong \frac{1}{2}m_h^* + \frac{1}{2}m_l^*$ (for details see discussion in Supplementary Material, Section F & G). Substituting $m^*(\text{MB})$ and m_l^* with the above measured values, we get $m_h^* = 2.5 \pm 0.5 m_e$ and $1.7 \pm 0.2 m_e$ in the cases a) and b), respectively. Again, the m_h^* deduced in the case a) is in better agreement with the values given by previous theoretical calculations [25] and experimental measurements [11]. Therefore we conclude that the case a) is the scenario most consistent with the experimental observations.

Figure 4(b) shows the schematic energy band structure based on the case a). Assuming a parabolic dispersion relation, the Fermi energy E_F is calculated to be ~ 8.0 meV, and the separation between the light and the heavy subbands is ~ 5.5 meV at the Γ point. We note that for the occurrence of magnetic breakdown the inequality $\hbar e B / 2\pi m^* \geq E_g^2 / E_F$ must be satisfied, where E_g is the energy difference between the two subbands at the tunneling point [26,32]. This criterion suggests that the heavy subband should have a steeper dispersion relation in the k_{MB} direction than the light subband. We note that since spin-orbit coupling is a comparable scale to E_F , it should have significant impact on the dispersion relationship discussed here, particularly at the tunneling points (see also discussion in Supplementary Material, Section H, considering a dominant Rashba-coupling scenario).

5. Conclusions

We have demonstrated well-resolved SdH quantum oscillations and Hall plateaus in the q2DEG confined at the $\text{LaAlO}_3/\text{SrTiO}_3$ interface in the low density regime. We found that the indices of the Hall plateaus have an interval of ~ 4 , and that the high- and low-field frequencies of the SdH

oscillations have a ratio of ~ 3 . In a magnetic breakdown scenario, this 4-fold degeneracy quantitatively accounts for all the mobile carriers in the observed quantum transport.

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Appendix

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Figure captions

Fig. 1. Quantum oscillations in magnetoresistance. R_{xx} and R_{xy} as a function of B in (a) sample A, (b) sample B, (c) sample C, and (d) sample D. The thin lines are linear fits to the low field ($B < 5$ T) R_{xy} data. In the inset of each panel the Hall conductance, G_{xy} , as a function of B is shown in unit of e^2/h . The indices label the integer positions of e^2/h . The lower inset of (d) shows a photograph of a typical sample.

Fig. 2. Positions of plateaus in G_{xy} . The corresponding G_{xy} (in unit of e^2/h) at local maxima in dG_{xy}/dB data of (a) sample A, (b) sample B, (c) sample C, and (d) sample D. The numbers in the parentheses indicate the closest integer indices, as labeled in the insets of Fig. 1. The thin lines are guides for the eye.

Fig. 3. Fourier transform (FT) and fan diagram analysis of quantum oscillations. The oscillations are extracted from the magnetoresistance data of sample A shown in Fig. 1(a). (a) dR_{xx}^2/dB^2 and dR_{xy}/dB as a function of $1/B$. (b) FT of the data shown in (a). (c) Index of extrema (integer n for maximum and $n + 1/2$ for minimum) in dR_{xx}^2/dB^2 as a function of $1/B$. (d) Index of extrema in dR_{xy}/dB (integer n for maximum and $n + 1/2$ for minimum.) as a function of $1/B$. In (c) and (d) the indices are chosen to make the intercept of S_H between 0 and 1.

Fig. 4. Schematic electronic orbits. (a) Fermi surface when the q2DEG consists of one light (l) and one heavy (h) subband, showing the inner light circle and the outer heavier star-shaped

geometry. The dark and shaded MB1 and the yellow dashed MB2 indicate two possible magnetic breakdown (MB) orbits. The green dots indicate the MB tunneling paths. By symmetry there are 4 equivalent MB1 orbits and 2 for MB2. (b) Energy *vs* momentum dispersion along the k_x and k_{MB} directions for the l and h subbands.

Table 1. Structure and electronic properties of samples. The width (W) of Hall bar and the thickness of LaAlO₃ film (t_{LAO}). In the calculation of electron density, S_H and S_L are from the analysis of dR_{xx}^2/dB^2 ; the corresponding values from the analysis of dR_{xy}/dB are shown in the parenthesis. Note that the tabulated values assume no degeneracy factor.

Sample	W (μm)	t_{LAO} (uc)	Surface treatment	Electron density ($\times 10^{12} \text{ cm}^{-2}$)			μ ($\text{cm}^2 \text{ V}^{-1} \text{ s}^{-1}$)	Effective mass m^* (m_e)	
				n_{Hall}	eS_H/h	eS_L/h		$\sim 11 \text{ T}$	$\sim 4 \text{ T}$
A	10	8	None	4.7	1.45 (1.24)	0.48 (0.45)	7,600	1.30 ± 0.04	0.9 ± 0.1
B	5	10	CAFM	7.5	1.59 (1.64)	0.58 (0.58)	7,100	1.32 ± 0.03	-
C	5	5	CAFM	5.1	1.44 (1.38)	0.52 (-)	5,500	1.19 ± 0.03	0.72 ± 0.04
D	10	10	Cap 140 nm a-LAO	4.8	1.28 (1.23)	0.55 (-)	10,000	-	-

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