Observation of the quantum Hall effect in $\delta$-doped SrTiO$_3$

Y. Matsubara$^{1,2}$, K.S. Takahashi$^{1,3}$, M.S. Bahramy$^{1,4}$, Y. Kozuka$^4$, D. Maryenko$^1$, J. Falsón$^4$, A. Tsukazaki$^2$, Y. Tokura$^{1,4}$ & M. Kawasaki$^{1,4}$

The quantum Hall effect is a macroscopic quantum phenomenon in a two-dimensional electron system. The two-dimensional electron system in SrTiO$_3$ has sparked a great deal of interest, mainly because of the strong electron correlation effects expected from the 3$d$ orbitals. Here we report the observation of the quantum Hall effect in a dilute La-doped SrTiO$_3$-two-dimensional electron system, fabricated by metal organic molecular-beam epitaxy. The quantized Hall plateaus are found to be solely stemming from the low Landau levels with even integer-filling factors, $n = 4$ and 6 without any contribution from odd $n$'s. For $n = 4$, the corresponding plateau disappears on decreasing the carrier density. Such peculiar behaviours are proposed to be due to the crossing between the Landau levels originating from the two subbands composed of $d$ orbitals with different effective masses. Our findings pave a way to explore unprecedented quantum phenomena in $d$-electron systems.
Conventional semiconductors such as Si, GaAs and ZnO are the main workhorses in the studies of integer and fractional quantum Hall effects (QHEs)\(^1,4\). The mobile carriers in these materials are located in bands composed mainly of s and p orbitals. In contrast, the conduction band of perovskite transition metal oxides such as SrTiO\(_3\) (STO) is composed of 3d \(t_{2g}\) orbitals with a strong directional anisotropy\(^2\). When confined into a two-dimensional (2D) environment, these states can show very interesting properties\(^6,8\). At sufficiently high carrier densities, the \(t_{2g}\) conduction band is quantized into a ladder of light and heavy subbands, whereas at low carrier densities resulting subbands are dominated by heavy orbitals, \(d_{xz}/d_{yz}\). In the latter case, electron–phonon effects combined with the many body interactions could further modify the dispersion of subbands thereby leading to formation of unusual electron liquid states\(^9\). Consequently, the STO-based two-dimensional electron system (2DES) can exhibit a variety of unconventional quantum effects\(^10\). Moreover, since STO is a widely used substrate for epitaxial growth of versatile materials with exotic properties such as high-\(Tc\) superconductivity, ferroelectricity, ferromagnetism and topological phases\(^11,12\), one can potentially incorporate a STO-based 2DES with high mobility into such systems to realize novel quantum effects.

Owing to the recent progress of thin-film growth technique, the electron mobility of three-dimensional carriers of STO has reached 53,000 cm\(^2\) V\(^{-1}\) s\(^{-1}\) in single crystalline films\(^{13}\), which is larger than 22,000 cm\(^2\) V\(^{-1}\) s\(^{-1}\) of bulk single crystals\(^{14}\). However, preserving a metallic state with reasonably high mobility has proven to be a challenge when the carriers are confined in two dimensions. While there is a large number of studies on STO-based 2DES including LaAlO\(_3\)/STO (LAO/STO) interface and \(\delta\)-doped STO\(^{7,8,15–20}\), the realization of the QHE in this class of systems has proven to be elusive and, thus, yet to be demonstrated. In particular, the low mobility of the doped carriers and their high concentration have hindered the successful demonstration of the QHE in STO. Any attempt to reduce the carrier density below \(3 \times 10^{12} \text{cm}^{-2}\) has turned out to result in a non-metallic state\(^{15}\). Although with the recent advancement in the growth of LAO/STO interfaces one can now realize 2DES’s with a relatively low carrier density (in the range of \(10^{12} \text{cm}^{-2}\)) with maintaining a high mobility (nearly \(10,000 \text{cm}^2 \text{V}^{-1} \text{s}^{-1}\)), it is still practically impossible to reach Landau levels with filling factors \(\nu \leq 10\) (refs 17–20). The realization of QHE at low enough filling factors in an easily accessible magnetic field (\(~10 \text{T}\)) imposes the restriction on the carrier density, which favourably should be below \(1 \times 10^{12} \text{cm}^{-2}\). At higher carrier densities, it is therefore practically only possible to observe the Shubnikov-de Haas (SdH) effect, which is of course a different quantum phenomena from the QHE.

Here we employ molecular-beam epitaxy (MBE) at a very high temperature (1,200 \(^\circ\)C) with metal organic (MO) precursors (MOMBE)\(^{21}\) to grow STO heterostructures confining the 2DES and reach electron mobility exceeding \(20,000 \text{cm}^2 \text{V}^{-1} \text{s}^{-1}\) at charge-carrier densities below \(1 \times 10^{12} \text{cm}^{-2}\). With such a high mobility-low carrier density heterostructure, we can successfully reach the quantum Hall regime in STO. The 2DES shows clear signatures of the QHE but there are peculiar features; the quantization occurs only at even integer states (\(\nu = 4, 6\)) and the \(\nu = 4\) state disappears at low carrier concentration. To elucidate the origin for those, we performed first principles calculations and found that these features can be modelled if the spin susceptibility is small compared with the Landau level broadening and the crossover of hybridized two orbitals made of \(d_{yz}/d_{xz}\) is taken into account.

### Results

**Transport properties of \(\delta\)-doped SrTiO\(_3\).** The \(\delta\)-doped STO, studied here, is composed of a 100-nm-thick bottom STO buffer layer, a 10-nm-thick STO doped with \(3 \times 10^{19} \text{cm}^{-3}\) La, and a 100-nm-thick top STO capping layer epitaxially grown on a (001) STO single-crystal substrate. The device is sketched in Fig. 1a and its corresponding optical microscope image is shown in Fig. 1b. The sample is scratched by a needle to pattern a van der Pauw type device with size 500 \(\times\) 500 \(\mu\)m\(^2\). At the four edges of the scratched square, aluminium wire is ultrasonically bonded to make an Ohmic contact with the 2D layer. The longitudinal resistance \(R_{xx}\) and Hall resistance \(R_{xy}\) showing below are all raw data of one configuration in van der Pauw geometry without calculating the average with the orthogonal configuration value of \(R_{xx}\) and \(R_{xy}\). The sample is fixed with silver epoxy on a chip carrier with a metallic surface. Due to a large dielectric constant of STO at low temperature\(^{22}\), the chip carrier surface acts as a global back gate for STO-based 2DES and thus enables \(\text{in situ}\) tuning of carrier density. First, the as-grown samples, that is, without applying the back-gate voltage \(V_{G}\), are characterized at 2 K. Figure 1b shows the relation between the ‘2D’ mobility and the carrier density at 2 K for various \(\delta\)-doped STO samples and STO-based heterostructures that indicate pure electron conduction. The data are picked up from previous reports showing clear evidence of 2D conduction with low carrier density such as SdH oscillations in tilted magnetic field\(^{15–20}\). The quantum transport measurements are performed at dilution refrigerator temperatures and in magnetic fields up to 14 T.
employing a low-frequency (7–9 Hz) lock-in technique with a low excitation current of 100 nA to suppress heating.

The best demonstration of the QHE is achieved using a sample which becomes insulating below 1 K on floating the back-gate electrode. The application of a positive $V_{G}$ accumulates the charge carriers in the $\delta$-doped region and the device becomes conducting for $V_{G} > 4.3$ V. Figure 2a,b shows $V_{G}$ dependence of carrier density and mobility of the device at 50 mK. As $V_{G}$ increases, the carrier density, as determined from the Hall effect measurement, increases from $7.7 \times 10^{11}$ to $1.2 \times 10^{12} \text{cm}^{-2}$ (see Supplementary Note 1), roughly following the linear relationship. The slope of $2.8 \times 10^{11} \text{cm}^{-2} \text{V}^{-1}$ depicted as broken line corresponds to the model of a plane-parallel capacitor assuming a dielectric constant $\varepsilon = 20,000$ for the gate insulator STO and a substrate thickness of 500 $\mu$m. The mobility shows a maximum value of $18,000 \text{cm}^{2} \text{V}^{-1} \text{s}^{-1}$ at a carrier density of $1.0 \times 10^{12} \text{cm}^{-2}$ (see Fig. 2b) and thus is comparable with the value obtained in metallic as-grown devices shown in Fig. 1b. Figure 2c,d shows an example of the $R_{xx}$ and $R_{xy}$ at $V_{G} = 50$ mK for $V_{G} = 5.0$ V ($1.2 \times 10^{12} \text{cm}^{-2}$). First, one recognizes instantly the strong oscillations of $R_{xx}$ whose well-developed $R_{xx}$ minima coincide with the Hall plateau structures of $R_{xy}$. Second, the plateaus at the negative field axis can be clearly assigned to Landau level filling factors $\nu = 4$ and 6 in $R_{xy} = h/2e^{2}$. At the positive field axis, $R_{xy}$ is also well quantized for $\nu = 4$, whereas $\nu = 6$ deviates from the exact quantized value. Furthermore, Supplementary Note 2 shows that the $R_{xx}$ minima at integer-filling factors show thermally activated behaviour. Given the current state-of-the-art for STO heterostructures, of which quality is doubtless lower than that of the well-established high-mobility semiconductor heterostructures, the slight asymmetry in magnetotransport with respect to the magnetic-field direction is not very surprising. Despite all imperfections that the current structure may suffer from, for example, disorder, charge-carrier inhomogeneity, the all metrics mentioned above strongly suggests the realization of QHE in the $\delta$-doped STO.

To inspect the QHE in more detail, we acquired the magnetoresistance traces of $R_{xx}$ and $R_{xy}$ at various $V_{G}$'s and display them in Fig. 3. In accordance with expectations, when $V_{G}$ increases (that is, the carrier density increases), the positions of valleys and peaks in $R_{xx}$ (Fig. 3a) systematically shift to higher magnetic fields. However, a distinct behaviour is found for filling factors $\nu = 4$ and 6. While the state at $\nu = 6$ with a well-developed plateau at or close to $R_{xx} = h/6e^{2}$ (Fig. 3b) and $R_{xx}$ minima is observed for all $V_{G}$'s, the quantum Hall state at $\nu = 4$ strikingly vanishes, that is, $R_{xy}$ deviates from the quantized value and $R_{xx}$ minimum disappears, when $V_{G}$ is lowered. To visualize the quantization behaviour, Fig. 3c replots the data in the plane of $\sigma_{xx}(B)$ and $\sigma_{xy}(B)$ with $B$ being the parameter for various $V_{G}$'s. Such representation demonstrates that the curves seem to converge towards $(\sigma_{xy}, \sigma_{xx}) = (\pm 6e^{2}/h, 0)$, which seems to be a stable point for all $V_{G}$'s, while $(\pm 4e^{2}/h, 0)$ forms only at high $V_{G}$ (high carrier density). It should be noted that such conversions can be observed only when $R_{xy}$ plateau and $R_{xx}$ minima are realized simultaneously. It is quite evident that the observed quantization is imperfect since the $\sigma_{xx}$ deviates from its exact quantization value and $\sigma_{xy}$ does not reach zero. Such behaviour might be caused by an additional conduction channel or some small bulk conductance remaining even in the regime of the QHE, which does not show localization behaviour in the magnetic field. We believe that improving the sample quality and gaining more knowledge on the origin for the disorder in the heterostructure (among the suspects is the inactive La dopants) will result eventually in the exact quantization of $\sigma_{xy}(\nu^{2}/h)$ concomitant with $\sigma_{xx}$ reaching zero. Finally, we note that a conspicuous absence of odd filling factors at all $V_{G}$'s indicates either the spin degeneracy or the orbital degeneracy at high magnetic fields. Since our density functional theory (DFT) calculation presented below rules in (out) the former (latter), we expect that the Landau levels at high magnetic field are spin degenerate. This expectation may also be valid when the Zeeman spin splitting ($g\mu_{B}B$, where $g$ is the electron $g$-factor) is smaller than the other energy scales such as that arising from the disorder.

**Electronic structures of $\delta$-doped SrTiO$_{3}$.** To shed light on the mechanism of quantum oscillations, we have calculated the electronic structure of a $\delta$-doped STO thin-film sandwiched between two sufficiently thick undoped STO slabs using realistic tight-binding supercell calculations, incorporating the band-bending potential in the $\delta$-doped region (see Methods). To be consistent with the design of our experimental heterostructure, the thickness of the quantum well (QW) is considered to be 10 nm, as schematically shown in Fig. 4e. We have assumed a square potential well (Fig. 4f) and varied its depth until the total amount of carrier density confined inside, and in the vicinity of QW, becomes $n = 1.0 \times 10^{12} \text{cm}^{-2}$. Under these conditions, the QW formed at the $\delta$-doped region confines two subbands. As shown in Fig. 4g,h,i by the false colour scale, both subbands are dominantly made of heavy $d_{xz/yz}$ orbitals at the Fermi level $E_{F}$, whereas the $d_{yz}$ orbital is the main contributor at the bottom of the lowest subband. It is to be noted that these subbands are distinct from the subbands previously observed at the surface of STO and KTaO$_{3}$ (refs 6,23). In those systems, the near surface band-bending potentials are much deeper but effectively confined within a much narrower region (that is, a few STO units). Consequently, $d_{yz}$ orbitals contribute dominantly to the lowest subbands thereby making them highly dispersive, whereas the $d_{xz/yz}$ can only contribute to the heavy subbands at much higher energies near the Fermi level. In the present system, on the other

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**Figure 2** Transport properties of two-dimensional electrons in $\delta$-doped SrTiO$_{3}$. (a,b) Carrier density (a) and mobility (b) as a function of gate voltage $V_{G}$ at 50 mK. The slope depicted as broken line, $2.8 \times 10^{11} \text{cm}^{-2} \text{V}^{-1}$, denotes the calculated one for the carrier density against gate voltage, assuming a STO gate dielectric constant $\varepsilon = 20,000$. (c,d) Longitudinal resistance $R_{xx}$ (c) and Hall resistance $R_{xy}$ (d) versus magnetic field $B$ measured at 50 mK with $V_{G} = 5.0$ V. Horizontal dotted lines in (d) are the Landau level filling factors ($\nu$) defined as $R_{xy} = h/2e^{2}(\nu = 4, 5, 6)$.
Figure 3 | Carrier density dependence of quantum transport in δ-doped SrTiO$_3$. (a,b) Longitudinal resistance $R_{xx}$ (a) and Hall resistance $R_{xy}$ (b) versus magnetic field $B$ measured at 50 mK with various $V_G$’s. For $R_{xx}$, the traces are shifted vertically for clarity as denoted by horizontal bars. Horizontal dotted lines in b are the Landau level filling factors ($\nu = 2 - 6$). (c) Parametric plots of $(\sigma_{xy}(B), \sigma_{xx}(B))$ for various gate voltages at 50 mK. (d) A schematic of Landau levels as a function of magnetic field. Green and purple lines correspond to the Landau levels ($N$) of outer (EB$_1$) and inner (EB$_2$) subbands, respectively. All lines contain up and down spin states, assuming spin degeneracy due to the small $g$-factor. The thick blue and orange lines illustrates the chemical potentials ($\mu_1$ and $\mu_2$) for low and high $V_G$’s. If the chemical potential (blue line $\mu_1$) is located at the crossing point of $N = 1$ of the outer subband and $N = 0$ of the inner subband (indicated by the arrow), then $\nu = 4$ is expected to vanish. Shifting the chemical potential upward away from this crossing point (exemplified by the orange line ($\mu_2$)), $\nu = 4$ can appear. (e) Landau level arrangement when $\nu = 4$ disappears. $\Delta_2$ is the Landau level broadening. $E_{\text{DOS}}^\text{IFS}$ and $E_{\text{DOS}}^\text{IFS}$ are the cyclotron energies of the outer and inner FSs, respectively. Note that each Landau level $N$ is spin degenerate. (f) Landau level arrangement when $\nu = 4$ can be observed.

The SdH oscillations are also analysed experimentally. To properly determine the positions of peaks and valleys in $R_{xx}$ for the small amplitude oscillations at low fields, we take $d^2R_{xx}/dB^2$ and plot it as a function of $1/B$ at $V_G = 4.7$ V (see Fig. 4a). We then assign the integer indices to the $d^2R_{xx}/dB^2$ peaks (valley positions of $R_{xx}$), denoted by the closed circles in Fig. 4b and the half integer indices to the $d^2R_{xx}/dB^2$ valleys ($R_{xx}$ peaks), indicated by the open circles in Fig. 4b. The frequency is deduced from the slope of indices versus $1/B$. The change of the slope at $1/B = 0.3$ T$^{-1}$ ($B = 3.3$ T) from 12.9 T at high field to 6.4 T at low-field region signals two transport regimes.

**Discussion**

These two values agree quite well with the respective band calculation data as shown above. While the fact that the ratio of slope change is close to two may be interpreted as the spin degeneracy lifting at high field, this change turns out to be due to the peculiar subband structure of the present STO 2DES (see Supplementary Note 3). Taking the spin degeneracy into account, the total carrier density extracted from these two SdH frequencies (6.4 and 12.9 T) is found to be $9.0 \times 10^{11}$ cm$^{-2}$ (3.0 $\times 10^{11}$ cm$^{-2}$ for 6.4 T and 6.0 $\times 10^{11}$ cm$^{-2}$ for 12.9 T); slightly lower than the carrier density estimated from the Hall effect, $1.0 \times 10^{12}$ cm$^{-2}$. This deviation is much smaller than that of previous reports$^{8,16}$ and is likely due to a minor contribution from an additional conduction channel as mentioned above, which can only affect...
decomposed charge-carrier density distributions (denoted by EB1 for outer FS and EB2 for inner one) are plotted in closed (open) circles. The slope changes from 12.9 to 6.4 T around 1/(sandwiched between two sufficiently large slabs of undoped STO as shown in theoretically derived are indicated by horizontal dotted lines. (projected band structure of QW. (Rxx the Hall effect (and not the SdH oscillations). This may shows non-vanishing values in its limit, and thus can no longer contribute to the oscillatory part of oscillations for Rxx/IFS (as denoted by the dashed lines in Fig. 4c,d). The Fermi surface formed by the confined subbands. We have also calculated the cyclotron effective mass for each FS (m^*_{IFS} and m^*_{0FS}; the cyclotron effective mass for outer and inner FS) using the relation \( m^* = \frac{h^2}{2m_0} \left| \frac{\partial^2 R_{xx}}{\partial B^2} \right| \) and obtained m^*_{0FS} = 1.17 m_0 and m^*_{IFS} = 0.62 m_0. To compare these values with the experiment, we have deduced m^* from the temperature dependence of the SdH oscillations after subtracting the non-oscillating background (\( \Delta R_{xx} \), Fig. 4c). To be consistent with our calculations, we consider the Rxx oscillations for n = 1.0 × 10^{12} cm^{-2} corresponding to \( V_G = 4.7 \) V. m^* is then determined at each \( \Delta R_{xx} \) extremum, as denoted by the dashed lines in Fig. 4c,d (additional information is provided in the Supplementary Note 4). Because of the weighted contribution of large and small FSs to Rxx, the electron mass at low fields is clearly smaller than that at high fields despite the uncertainty in estimated values of m^* (indicated by the error bars in Fig. 4d). This tendency is in accordance with our calculations of m^*_{IFS} and m^*_{0FS}, predicting a smaller (larger) m^* for the inner (outer) FS as indicated by horizontal lines.

Such a mixed subband contribution strongly affects the appearance of the QHE. Taking into account the relative positions of the subbands and their different effective masses, a schematic fan diagram for the spin-degenerate Landau levels stemming from each subband is depicted in Fig. 3d; green and purple lines correspond to the Landau levels (N) of outer (EB1) and inner (EB2) subbands, respectively. This diagram can explain both the disappearance of v = 4 and the stability of v = 6. As pointed out in refs 24, 25, an even integer-filling factor is pointed out in refs 24, 25, an even integer-filling factor is...
in this situation, $E_B$ is at filling factor $\nu = 3$ and $E_B^2$ is at $\nu = 1$, so that the total filling factor becomes 4. However, this does not lead to the QHE, since $\mu_1$ is not in a gap. Changing the charge-carrier density, and correspondingly relative population of the subbands, can shift the chemical potential into the gap between the Landau levels and thus lead to the formation of QHS at $\nu = 4$ as illustrated in Fig. 3f. In the same manner, we can explain the stability of QHS at $\nu = 6$. Considering the fact that $E_B$ and $E_B^2$ have different energies, the QHS at $\nu = 6$ can be suppressed if the filling factors of these subbands are $\nu = 5$ and $\nu = 1$, respectively. This, however, imposes a large imbalance on the charge-carrier densities of subbands, which cannot practically be realized in our STO structure. Thus, the QHS at $\nu = 6$ is found to be stable in our experiment. In addition, one can find that slightly higher chemical potential than $\mu_2$ makes $\nu = 4$ and 8 quantum Hall plateau more stable. However, much higher magnetic field will be needed to observe the $\nu = 2$ plateau in that case.

At higher, we have observed QHE in $\delta$-doped STO grown at high temperature by MOMBE. This is the first observation of the QHE in perovskite oxides. The Hall conductance is quantized at even integer filling factors. The absence of odd integer filling factors is proposed to be due to the small anisotropy of subbands in the QW formed at the polar surface of the strong spin-orbit perovskite KTaO$_3$.

**Methods**

**MBE growth.** All films were grown by metal organic gas source molecular-beam epitaxy (MOMBE) at a high temperature [18,21]. In this method, Sr and La flux were evaporated from a conventional effusion cell with a pure elemental source, and 4.5 MBM growth was performed at a substrate temperature of 1,200°C and a beam equivalent pressure (BEP) of 8×10$^{-12}$ and 3×10$^{-12}$ cm$^{-2}$ as shown in Supplementary Note 1. Such a variation might be caused by several reasons. As shown in Supplementary Note 1, Supplementary Fig. 1f, one is the experimental uncertainty of actual La beam flux and activation ratio of dopant. In fact, the carrier density at room temperature is found to vary between 1.5×10$^{13}$ and 4.5×10$^{13}$ cm$^{-2}$. Another reason is a partial freezing of charge carriers while lowering the temperature. We found this freezing is more pronounced for samples with smaller carrier density at room temperature. This expands the variation of carrier density at 2 K towards smaller carrier density side. The reason of partial freezing is not clear but such a behaviour has been commonly observed in a number of previous studies on STO-based 2DESs [18]. Taking into account the fact that thick single crystalline films with comparable or smaller La concentration do not show such a behaviour [18], we presume that the charge-carrier freezing is related to the localization of carriers due to disorder effect pronounced by confinement. Distilled pure ozone as oxidizing agent was generated and supplied from MPOG-Ci $n$/P$^1-0$.

**Electronic structure calculation.** To calculate the interface band structure, we initially performed a DFT calculation using the Perdew–Burke–Ernzerhof exchange–correlation functional within Density Functional Theory (DFT) with the projector augmented wave (PAW) basis set [28]. The results of DFT Hamiltonian was then down-folded using maximally localized Wannier functions to generate a 200 unit cell tight binding supercell stacking along [001] direction with additional on-site terms, accounting for the QW potential. The same method has been already applied and successfully reproduced the results of ARPES data of the QHE confined at the surface of STO [29]. Assuming a 10-nm-thick interface, the depth of QW was varied until the total amount of carrier density confined at, and in the vicinity of, the interface became $n = 1.0 \times 10^{12}$ cm$^{-2}$.

We emphasize that in our tight-binding supercell Hamiltonian, there is no adjustable parameter other than an on-site potential term, representing the band-bending potential in the QW. Even for this bending potential we consider the same width as that realized in our experiment. The only variable parameter in our calculation, as mentioned above, is the depth of the potential that is chosen such that it yields the same confined charge-carrier density as that observed in our experiment ($1.0 \times 10^{12}$ cm$^{-2}$).

**Data availability.** The authors declare that the data supporting the findings of this study are available within the article and its Supplementary Information.

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Author contributions

Y.M. and K.S.T. grew and characterized the films. Y.M., Y.K., and J.F. performed the low-temperature measurements. M.S.B. performed the electronic structure calculations. Y.M. and K.S.T. analysed the data. D.M., A.T., Y.T. and M.K. contributed to discussion of the results and guided the project. Y.M., K.S.T., M.S.B. and D.M. wrote the manuscript with contributions from all authors.

Additional information

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