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Two-dimensional superconductivity and anisotropic transport at KTaO_3 (111) interfaces

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The unique electronic structure found at interfaces between materials can allow unconventional quantum states to emerge. Here we report on the discovery of superconductivity in electron gases formed at interfaces between (111) oriented KTaO_3 and insulating overlayers of either EuO or LaAlO_3 . The superconducting transition temperature, approaching 2.2 K, is about one order of magnitude higher than that of the $\text{LaAlO}_3/\text{SrTiO}_3$ system. Strikingly, similar electron gases at KTaO_3 (001) interfaces remain normal down to 25 mK. The critical field and current-voltage measurements indicate that the superconductivity is two dimensional. In EuO/KTaO_3 (111) samples, a spontaneous in-plane transport anisotropy is observed prior to the onset of superconductivity, suggesting the emergence of a distinct 'stripe' like phase, which is also revealed near the critical field.

Superconductivity in two dimensions (2D) has been a central theme in condensed matter physics and material science (1). In 2D, the electron-electron and electron-lattice interactions that mediate pairing can also give rise to states that compete with superconductivity, and fluctuations associated with reduced dimensionality can further suppress superconductivity. Thus, although there are many examples of two-dimensional electron gases (2DEG) and ultrathin metallic films, only a small fraction of these are superconducting. Most of the foundational work in 2D superconductivity was carried out using amorphous thin films (2), and led to deep insights regarding the nature of classical and quantum phase transitions in the 2D limit. In more recent years, there have been striking developments in the realization of 2D superconductivity in crystalline materials (1), including layered transition metal chalcogenides (3, 4), bilayer (5) and trilayer (6) graphene, gated surfaces of single crystals (7, 8), atomically thin ordered metallic layers on semiconductor surfaces (9), and interfaces between crystalline materials (10–12). These crystalline 2D superconductors allow us to realize and break symmetries, and tailor electronic structure in ways that are not possible in amorphous and disordered thin films. For example, in a 2D superconductor with strong spin-orbit coupling and broken inversion symmetry, a Rashba interaction may lead to a superconducting order parameter with mixed *s*-wave and *p*-wave symmetry (13), a candidate platform for

realizing Majorana modes (14). It is perhaps telling that three of the most prominent examples of 2D superconductivity at crystalline interfaces – $\text{LaAlO}_3/\text{SrTiO}_3$ (LAO/STO), $\text{FeSe}/\text{SrTiO}_3$ and $\text{La}_2\text{CuO}_4/\text{LaSrCuO}_4$ – involve transition metal oxides (10–12), where strong electron-electron and electron-lattice interactions likely play a role in mediating superconducting pairing. In some of these interfacial systems, the superconductivity is believed to be 'unconventional' – i.e., with a non *s*-wave order parameter, and a pairing mechanism that is not described by the Bardeen-Cooper-Schrieffer (BCS) theory.

KTaO_3 (KTO) is an insulator with a cubic perovskite structure, a bandgap of 3.6 eV, and a dielectric constant that grows to > 4500 upon cooling to low temperatures (15, 16). It is believed that KTO is on the verge of a ferroelectric transition, thwarted by quantum fluctuations at low temperatures. It is thus called a 'quantum paraelectric', much like SrTiO_3 (STO). STO can be easily doped into a metallic state that becomes superconducting at $T < 0.5$ K (17). In contrast, few studies on doped KTO exist for bulk samples (18, 19), and none of them report any signatures of superconductivity. Nonetheless, it was reported that ionic liquid gating could be used to tune the KTO (001) surface into a weak superconducting state with $T_c \sim 47$ mK (8). The electronic states near the Fermi level in electron doped KTO are derived from Ta-5*d* states – in particular d_{xy} , d_{zx} and d_{yz} orbitals (20). The large spin-orbit

coupling in KTO lifts the degeneracy of these states and splits them into $J = 1/2$ (where J is the total angular momentum) and $J = 3/2$ states, with the former being ~ 0.4 eV higher in energy (compared to ~ 0.025 eV in STO). The electrons in KTO occupy the lower energy $J = 3/2$ states which are degenerate near the Γ -point and disperse as 'light' and 'heavy' electron states at finite momenta. Unlike in STO, there are no symmetry lowering lattice distortions at low temperatures to lift this degeneracy at the Γ -point, though confinement can also lift degeneracies. 2DEGs can be realized at the KTO (001) and (111) interfaces via vacuum cleaving (21, 22) followed by exposure to UV or synchrotron radiation. Angle resolved photoemission spectroscopy (ARPES) studies on the KTO (111) surface found a distinct Fermi surface with a six-fold symmetry derived from the lattice structure (Fig. 1, A and B), spin split-off bands, and evidence for band bending and confinement near the surface (21, 23). Owing to the large spin-orbit coupling, the interfacial electron gas at KTO (111) interfaces (24, 25) is also expected to host complex spin textures in momentum space. In the particular case of a bilayer of KTO along the [111] direction, which forms a buckled honeycomb lattice (Fig. 1B), calculations suggest that this system may support topological states (26, 27).

Here, we report on the observation of 2D superconductivity in electron gases formed at oxide-insulator/KTO (111) interfaces. We have measured T_c as high as 2.2 K, and this value can be tuned by varying carrier density during the sample growth. We also observe an emergent stripe phase which breaks the rotational symmetry in the KTO (111) surface.

The 2DEGs on KTO studied in this work were prepared by growing an overlayer of EuO with molecular beam epitaxy (MBE), or LAO using pulsed laser deposition (PLD) (28). We characterized the chemical oxidation state of the EuO/KTO (111) interface using X-ray absorption near-edge spectroscopy (XANES) at the Ta- L_{3s} edge (9.88 keV). XANES measurements were carried out adopting two different angles of incidence, one close to grazing incidence (0.16°) that probed only the first few nanometers of the KTO near the EuO/KTO interface, and another at a higher angle (5.16°) that probed the bulk of the KTO. As shown in Fig. 1C, these measurements indicate that the Ta oxidation state near the surface is very close to that in the bulk (Ta^{5+}), and subtle difference in the absorption curves suggests that the interfacial Ta is only slightly reduced. In particular, none of our measurements show any evidence for metallic Ta, which is known to be superconducting in ultrathin films (29). Shown in Figs. 1, D and E, are scanning transmission electron microscopy (STEM) measurements of our samples, which indicate abrupt interfaces between an annealed KTO (111) surface and the polycrystalline EuO or amorphous LAO overlayer, as confirmed by X-ray diffraction measurements. Using aberration-corrected high-resolution TEM and scanning TEM, we find oxygen vacancies near the

EuO/KTO (111) interface, and Eu substitution on the K sites within the first three unit-cells (fig. S1), both of which can dope the interfacial KTO with electrons. Similar doping effects are also observed at the LAO/KTO (111), EuO/KTO (001) and LAO/KTO (001) interfaces (figs. S2 and S3).

Figure 2A shows transport measurements on 2DEGs in EuO/KTO (111) and (001) interfaces, both of which display metallic behavior from 300 K to 4 K. As temperature is lowered further, the EuO/KTO (111) interface shows superconductivity, which is presented in Fig. 2B with four samples. These samples were grown at different temperatures and oxygen pressures, resulting in different carrier densities and mobilities (28). The T_c , determined at half of the normal state resistance R_N , reaches as high as 2.2 K in sample EuO/KTO(111)_1. This transition temperature is about one order of magnitude higher than those ($T_c \sim 200$ mK) found at the LAO/STO interfaces. To investigate whether the EuO overlayer is necessary for superconductivity, we have prepared 2DEGs using LAO overlayers. As shown in Fig. 1C, superconductivity with $T_c > 1.1$ K is also observed in LAO/KTO (111) samples, demonstrating that the emergence of superconductivity does not depend on a specific overlayer of the 2DEG.

We have also performed low-temperature measurements on 2DEGs formed at KTO (001) interfaces. Figure 2D shows the measurement results for a series of (001) samples including both EuO and LAO overlayers. The carrier density n_s of these samples varies from $5.8 \times 10^{13} \text{ cm}^{-2}$ to $1.1 \times 10^{14} \text{ cm}^{-2}$, covering the same range of densities as the (111) samples shown in Fig. 2, B and C, but slightly lower than the n_s values in Ref. (8). Unlike the (111) samples, no superconductivity is observed in these (001) samples down to 25 mK. This crystallographic orientation dependent interfacial superconductivity is in sharp contrast with 2DEGs at STO interfaces, where superconductivity, with about the same T_c , occurs for all orientations (10, 22, 30). Our STEM characterizations of the KTO (001) interfaces show similar interfacial chemical composition as the (111) samples. This leads to the possibility that the unique electronic structure in the KTO (111) surface may play a role in inducing strong Cooper pairing among the interfacial charge carriers, which calls for further investigation.

The superconductivity at the KTO (111) interface is two-dimensional. This is borne out by upper critical fields $B_{c\perp}$ and $B_{c\parallel}$ measured perpendicular and parallel to the KTO (111) plane, respectively, and defined at the half of R_N . Figure 3, A and B, show the magnetic field dependence of R_s measured on sample EuO/KTO(111)_3. The temperature dependence of $B_{c\perp}$ is well described by the Ginzburg-Landau (G-L) theory which yields a linearized equation: $B_{c\perp}(T) = \Phi_0(1 - T/T_c)/[2\pi(\xi_{GL})^2]$ where Φ_0 is the magnetic flux quantum and ξ_{GL} is the G-L coherence length at $T = 0$ K. The linear fit to $B_{c\perp}(T)$ in Fig. 3C gives $\xi_{GL} \approx 13$ nm. The extrapolated value of $B_{c\perp}(0) \approx 1.8$ T is $> 10^3$ times higher than the critical field

observed in ionic-liquid gated KTO (001) devices (8). Further, we find that the critical fields are highly anisotropic. For fields applied in the sample plane, $B_{c\parallel}$ is much higher than $B_{c\perp}$ with the ratio $B_{c\parallel}/B_{c\perp} > 10$ at $T = 0.6$ K (Fig. 3C). The temperature dependence of $B_{c\parallel}$ shows a characteristic square root dependence as expected from Tinkham's model (31): $B_{c\parallel}(T) = \Phi_0[12(1 - T/T_c)]^{1/2}/(2\pi d\xi_{GL})$, where d is the effective thickness of the superconducting state. From the fit to $B_{c\parallel}(T)$, we obtain $d \approx 5.1$ nm $< \xi_{GL}$, which is consistent with 2D superconductivity.

We note that $B_{c\parallel}(0) \approx 17.0$ T extrapolated from the fit is substantially larger than the paramagnetic pair-breaking field $B_P = \Delta_0/(2^{1/2}\mu_B) \approx 3.2$ T based on BCS theory in the weak-coupling limit (32, 33), where $\Delta_0 \approx 1.76k_B T_c$ is the zero-temperature superconducting gap, k_B is the Boltzmann constant and μ_B is the Bohr magneton. High values of $B_{c\parallel}$ in excess of B_P can be realized in the presence of spin-orbit coupling owing to elastic scattering (33, 34) in the limit where the mean free path $l_{mfp} \ll \xi_{GL}$, though other spin-relaxation mechanisms may apply even in the clean limit in the presence of broken inversion symmetry and high spin-orbit coupling (35). Using a single-band model where $l_{mfp} = \hbar/(e^2 k_F R_s)$, $\hbar$ is the Planck constant, e is the electron charge, and $k_F = (2\pi n_s)^{1/2}$ is the Fermi wavevector, our estimates for different samples suggest $l_{mfp} \sim 0.4$ to 0.7 times ξ_{GL} which is intermediate between the clean and dirty limits. Enhanced $B_{c\parallel}$ is also expected in superconductors that are believed to have a spin-triplet p -wave order parameter, though these were thought to be unstable in the presence of disorder. However, recent theoretical developments indicate that in the presence of strong spin-orbit coupling, criteria that might apply to KTO (111) interfaces such as broken inversion symmetry (36), spin-momentum locking (37) or multi-orbital superconducting states (38) can stabilize unconventional superconductivity against disorder scattering. Thus, at this time, the mechanism for realizing $B_{c\parallel}$ values in excess of B_P at KTO (111) interfaces remains an open question. In Fig. 3A, we also observe an approximate crossing point near $B_{\perp} = 2.1$ T, which implies the existence of a nearly temperature-independent separatrix in R_s vs. T traces that show either a positive or negative temperature coefficient of resistance upon cooling, depending on magnetic field (fig. S4). Such data may be interpreted in terms of a quantum phase transition between a 2D superconductor and a correlated metal (39).

The superconducting state in the EuO/KTO sample shows a robust critical-current behavior. The measured I - V characteristics at different temperatures are shown in Fig. 3D, where the critical current I_c approaches 200 μ A at $T = 1$ K. The I_c exceeds 200 μ A at lower temperatures (Fig. 3F). A lower bound of the critical current density, $K_c(1\text{ K}) = 500$ μ A cm $^{-1}$, is obtained by assuming the current spreads over the

entire width of the sample (4 mm). A more precise determination of K_c requires measurements using patterned Hall bar devices. As the temperature is raised to near T_c , we observe a gradual onset of a resistive state at low currents. In Fig. 3E we plot the I - V on a log-log scale, and observe that the slope of the I - V characteristics evolves smoothly from the normal Ohmic state, $V \propto I$, toward a steeper power law, $V \propto I^\alpha$, as superconductivity sets in at lower temperatures. This evolution may be interpreted in terms of a Berezinskii-Kosterlitz-Thouless (BKT) transition in a 2D superconductor (40), where the onset of a non-linear I - V characteristic in the superconducting state is caused by the current driven unbinding of vortex anti-vortex pairs that are created by thermal fluctuations at finite temperatures. This causes the exponent α to smoothly evolve from a value of 1 above and near the onset of superconductivity to higher values as the temperature is lowered (41, 42), with the $\alpha = 3$ at T_{BKT} . Red dashed line in Fig. 3E corresponds to a I^3 dependence, which indicates that the T_{BKT} of this sample EuO/KTO(111)_3 is between 1.2 K and 1.3 K, which is slightly below the zero-resistance T_{c0} of about 1.34 K. At temperatures below T_{c0} , we observe hysteresis in the I - V curves near I_c (Fig. 3F), which suggests that the 2D superconductivity is inhomogeneous, with superconducting regions joined by weak links (43).

In low carrier density EuO/KTO (111) samples, we observe the emergence of a distinct phase near the superconducting state, as revealed by a spontaneous in-plane anisotropy in resistance. Shown in Fig. 4A is the measurement in sample EuO/KTO(111)_5, with $n_s = 3.5 \times 10^{13}$ cm $^{-2}$ and a relatively high mobility of about 279 cm 2 V $^{-1}$ s $^{-1}$ at $T = 10$ K. The measurements are carried out in a van der Pauw geometry (Fig. 4B). As temperature is lowered below about 2.2 K (Fig. 4A), the resistance increases by almost 50% for current along the $[1\bar{1}0]$ crystal axis, whereas it decreases by over 50% for current flowing in the $[11\bar{2}]$ direction. We note that although the van der Pauw method can amplify the transport anisotropy, the qualitative feature reported here should be unaffected (44). A similar approach has been used in the past to characterize resistance anisotropy in high-mobility 2DEGs in the quantum Hall regime (45). The formation of this anisotropy in our samples suggests the emergence of a distinct phase (46) that breaks rotational symmetry over macroscopic length scales (fig. S5). The observed anisotropy persists over a broad temperature range from 2.2 K down to about 0.7 K, which is shown with a green background in Fig. 4A. At still lower temperatures, the resistance in both the $[1\bar{1}0]$ and $[11\bar{2}]$ current directions drop rapidly to zero, and the superconducting state is obtained (light blue region).

The increase in resistance along the $[1\bar{1}0]$ direction (Fig. 4A) upon lowering temperature from the normal state is similar to observations in 2D granular superconductors (47),

where superconducting puddles develop first. This occurs owing to the gapping out of quasiparticles by a superconducting gap (48), which inhibits transport between weakly coupled superconducting regions. Global superconductivity is restored at still lower temperatures presumably via Josephson coupling between these regions. The decrease in resistance along $[1\bar{1}2]$ at the same temperature suggests the possibility of stronger coherent coupling between the superconducting regions in that direction. This would naturally occur if the underlying superconductivity is anisotropic, making superconducting regions organize themselves into ‘stripes’ as illustrated in Fig. 4B. Our measurements also imply that the orientation of the stripes breaks the 3-fold rotational symmetry of the KTO (111) surface. A possible explanation is that these stripes are pinned by the sample edges, and remain coherently aligned over macroscopic length scales. We note that pre-formed superconducting puddles have also been suggested in the LAO/STO system (49).

The magnetic field dependence of sheet resistance provides further evidence for an anisotropic ‘stripe’ like phase. Figure 4C shows R_s measured in the same EuO/KTO(111)_5 sample in an out-of-plane magnetic field at $T = 0.1$ K, which is well below T_{co} . As the magnetic field is increased to near the critical field, there is a sharp rise in the resistance along both directions of current. The initial high/low resistance values along $[1\bar{1}0]/[11\bar{2}]$ in the resistive state are approximately equal to those in the ‘stripe’ phase shown in Fig. 4A, obtained by just raising the temperature to above T_{co} . This suggests that the ‘stripe’ phase is obtained by a suppression of global superconductivity upon the application of a magnetic field. By increasing the magnetic field further, the resistance decreases along $[1\bar{1}0]$ and increases along $[11\bar{2}]$, as the ‘stripe’ phase is driven into the normal state at $B_{\perp} \geq 0.8$ T where the resistance anisotropy also disappears, presumably thanks to a more complete suppression of the superconducting gap within the stripes.

Figure 4, D to F, shows magnetotransport measurements in other EuO/KTO (111) samples, with higher carrier densities and lower mobilities than that of EuO/KTO(001)_5. The resistance anisotropy, showing the same dependence on the crystal axes, is observed in all of these samples to varying degrees around the critical field. The magnitude of the transport anisotropy, normalized by the normal-state value, becomes progressively smaller as the mobility of the sample decreases. In sample EuO/KTO(111)_3, which has the lowest mobility among these samples, the transport is nearly isotropic (Fig. 4F). This suggests that the ‘stripe’ phase depends on the disorder and/or carrier density within the sample. We point out that in general, nematic or stripe like states are known to be very sensitive to disorder in 2D (50) – for example in the layered material $\text{Sr}_3\text{Ru}_2\text{O}_7$ (51) the stripe like phase

is only observed in very clean samples. Furthermore, the ‘stripe’ phase in our samples seems to not compete with the superconductivity. For example, sample EuO/KTO(111)_2 clearly shows ‘stripe’ like anisotropy (Fig. 4E), but it also has higher T_c than that of EuO/KTO(111)_3 (see Fig. 2B), which shows very weak anisotropy (Fig. 4F). We note that our limited transport measurements in LAO/KTO (111) samples, where the overlayer LAO is non-magnetic, show that the transport is mostly isotropic when an out-of-plane field is applied (fig. S6). However, a large anisotropy is observed upon the application of an in-plane magnetic field approaching $B_{c||}$. Our findings suggest that the ‘stripe’ phase may be controlled by the interaction between electron spins and an effective magnetic field – either via the exchange coupling with the magnetic EuO overlayer in EuO/KTO (111) samples, or the external in-plane magnetic field as for the case of LAO/KTO (111).

The 2D superconductivity observed in the KTO (111) interfaces shows substantially higher T_c than that in the widely studied LAO/STO system, and the T_c increases monotonically with n_s in the measured range. These higher T_c values make fundamental studies and device applications of superconducting 2DEGs using crystalline interfaces more readily accessible. The origin of the superconductivity is still an open question. Possible links between superconductivity and the large spin-orbit interaction in KTO (111) 2DEGs, as has been suggested in LAO/STO (111) (52, 53), could be explored in future work.

When global superconductivity is suppressed by either temperature or a magnetic field, transport measurements reveal a ‘stripe’ phase which produces large anisotropic transport in the (111) KTO plane. Notably, the directions for the high/low resistance states in the vicinity of superconductivity are oriented along the same crystal axes in the KTO and STO (111) interfaces (54, 55). We observe no appreciable dependence on thermal cycling across the onset temperature of the ‘stripe’ phase (fig. S7), as has been observed in LAO/STO (56), where these effects were caused by the formation of structural domain walls. Although our X-ray and STEM measurements carried out at 300 K do not indicate any structural distortions that would break the 3-fold symmetry of the KTO (111) surface, we cannot rule out weak distortions below our measurement resolution, or the emergence of such distortions at lower temperatures. In addition, the ‘stripe’ phase shows strong dependence on magnetic field, mobility and/or carrier densities, which suggests a magnetic and/or electronic origin. A possible explanation for the observed anisotropy may be a spatially modulated or inhomogeneous superconductivity. This can happen for example in a Fulde-Ferrel-Larkin-Ovchinnikov (FFLO) state, where a spin-split Fermi surface formed owing to a magnetic proximity effect or an external magnetic field resulting in Cooper pairs with

nonzero total crystal momentum (36, 46), whose density is periodically modulated in real space. It is also possible that the superconductivity at KTO (111) interfaces is inherently composed of a spatially modulated density of Cooper pairs or a pair density-wave (PDW) (57). Further experiments, including probes of the spatial structure of superconductivity, will be helpful for elucidating the nature of superconductivity and resistance anisotropy that we observe.

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SUPPLEMENTARY MATERIALS

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Materials and Methods
Figs. S1 to S8
Table S1

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Fig. 1. Schematics of KTO (111) surface, XANES and STEM characterizations. (A) KTaO_3 lattice structure. The relative sizes of the ions are chosen to emphasize the Ta atoms. The three adjacent (111) planes containing Ta^{5+} ions are colored in light purple, blue and green, respectively. (B) Distribution of Ta^{5+} ions viewed along the [111] crystal axis. Ta^{5+} ions are shown with progressively smaller sizes in the three adjacent (111) planes, which are labeled as Ta – I, Ta – II and Ta – III, respectively. Solid lines between Ta^{5+} ions indicate the relative distance or coupling strength – with thicker lines representing stronger couplings, giving rise to a buckled honeycomb lattice for the first bilayer comprising Ta - I and Ta - II sites. (C) XANES data from sample EuO/KTO(111)_4 at the Ta L-edge. The KTO near the surface is only slightly reduced, with Ta valence close to its bulk value (5+). XANES data for pure Ta is also shown for comparison. STEM images of the (D) EuO/KTO (111) and (E) LAO/KTO (111) interfaces, looking down the $[1\bar{1}0]$ direction. Green box indicates the region near the interface.

Fig. 2. Transport measurements of 2DEGs formed at different KTO interfaces. (A) Metallic temperature dependence of the sheet resistance of EuO/KTO (111) and (001) samples measured from 300 K to 4 K. (B) Measurement at lower temperatures shows superconducting transitions in EuO/KTO (111) samples (current along $[11\bar{2}]$) with varying carrier densities, which are determined from Hall measurement at $T = 10$ K for samples EuO/KTO(111)_1, 2 and 3. The carrier density in EuO/KTO(111)_4 is estimated from growth condition. (C) Similar measurements on LAO/KTO (111) samples also show superconductivity. (D) No superconductivity is observed in samples with (001) oriented KTO interfaces with overlayers of either EuO or LAO down to 25 mK. The range of the carrier density is similar to those of the (111) oriented samples shown in (B) and (C).

Fig. 3. Critical field and current-voltage measurements on sample EuO/KTO(111)_3. (A,B) Sheet resistance measured at different temperatures as a function of the out-of-plane and in-plane magnetic fields, respectively. (C) Temperature dependence of critical fields, which are determined at half of R_N (dotted horizontal line in (A) and (B)). (D) I - V curves measured at different temperatures. (E) I - V curves plotted on a logarithmic scale using same color codes as in (D). Black solid lines are linear fits to the data. Red dashed line is $V \propto I^3$, which is used to infer the BKT transition temperature. (F) Hysteresis of I - V curves near the critical current measured at temperatures below T_{c0} . In all the measurements (A)-(F), the current is applied along $[11\bar{2}]$ direction.

Fig. 4. Stripe phase measured in different EuO/KTO(111) samples. (A) Sheet resistance of sample EuO/KTO(111)_5 measured with electric current along $[1\bar{1}0]$ (red) and $[11\bar{2}]$ (blue) crystal axes under zero field. The light blue and green region indicate superconducting (SC) and 'stripe' state, respectively. (B) Illustration of the measurement geometry for the case of current (red arrow) along $[1\bar{1}0]$ direction perpendicular to the stripes. These stripes may be composed of Cooper pairs, which are shown in light blue bubbles. (C)-(F) Magnetic field dependence of the sheet resistance measured along both current directions at $T = 0.1$ K in samples with decreasing mobilities. Stripe phase is revealed in all samples around the critical field (green region). Note that EuO/KTO(111)_2 has a higher T_c than EuO/KTO(111)_3, but also shows a more prominent transport anisotropy.