Observation of transient superconductivity at LaAlO$_3$-SrTiO$_3$ interface

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The mutual interplay of point group symmetry, charge inversion symmetry, U(1) gauge symmetry and spin rotation symmetry in heterostructures of complex perovskite oxides lead to the co-existence of a host of intriguing properties - ferroelasticity, ferroelectricity, superconductivity and ferromagnetism [1, 2]. Superconductivity and magnetism are generally considered to be incompatible with each other and hence the observation of the co-existence of these two phases in the conducting electronic subsystem has not been observed in condensed matter systems.

Despite intensive research over the last decade [1] there is no clear understanding of the origin of superconductivity and ferromagnetism in this system. The breaking of mirror inversion symmetry at the interface lifts the degeneracy of the $t_{2g}$ levels of Ti ions at the interface [5] with the $d_{xy}$ level having lower energy than the $d_{xz}$ and $d_{yz}$ orbitals [6]. At low number densities all the conduction electrons occupy the lower lying $d_{xy}$ orbitals at the interface [1]. It has recently been proposed that when the number density $n$ of itinerant electrons exceeds a certain critical value the system undergoes a Lifshitz transition at which point the $d_{xz}/d_{yz}$ bands near the interface begin to get occupied. The system now effectively has two types of carriers - a high density electron gas residing in the $d_{xy}$ orbital and a lower density high moblility electron gas occupying the $d_{xz}/d_{yz}$ orbitals [7–9]. It is this second group of electrons that are believed to be responsible for superconductivity in these materials [10].

Our measurements were performed on samples with 10 unit cells of LaAlO$_3$ grown by Pulsed Laser Deposition (PLD) on TiO$_2$ terminated (001) SrTiO$_3$ single crystal substrates (details of the sample preparation and measurement techniques are in the methods section). The sheet resistance of the device as a function of temperature at different gate voltages $V_g$ is shown in figure 1(a). The superconducting transition temperature and the normal state resistance both depend sensitively on the gate voltage. The superconducting transition temperature (defined as the temperature where resistance drops to 50% of its normal state resistance) increases as the system is progressively electron doped and has a maxima of about 200 mK (see figure 1(b)) in conformity with previous observations in similar systems [8, 11, 12].

The magnetoresistance data for magnetic fields applied perpendicular to the interface measured at a few representative values of $V_g$ are shown in figure 2(a). The measurements were taken at 245 mK where the device is in the normal state at all measured gate voltages. We notice a distinct change in the nature of the magnetoresistance curves as $V_g$ changes from a large negative value to large positive value. Let us first consider the case when a large negative gate voltage $V_g = -200$ V is applied to the device (figure 2(b)). The magnetoresistance is negative, quite small in magnitude (about 4% at 8 T field) and is hysteretic. The hysteresis is time dependent and relaxes exponentially to an equilibrium value over a time scale of a few hundreds of seconds (details of the measurement of the relaxation time is provided in the Supplementary materials). Hysteresis in magnetoresistance has been seen previously in LAO/STO heterostructure devices and is taken to indicate the presence of ferromagnetic domains in the system [12, 13]. With increase in temperature the magnitude of hysteresis decreases and eventually vanishes by 1.5 K. With increasing $V_g$ the magnitude of hysteresis decreases and eventually vanishes at around a critical gate voltage $V_g \sim 110$ V (see figure 2(c)). We denote this value of gate voltage as $V_g^*$. The relaxation time also gradually decreases with increase in $V_g$. All this seems to indicate that as $n$ increases, the magnetic anisotropy energy decreases implying a gradual weakening of the ferromagnetic state with increased electron doping of the system.

For $V_g > V_g^*$, the magnetoresistance is positive as the magnetic field is swept from 0 T to 8 T. As the magnetic field is swept back down towards 0 T, the magnetoresistance curve retraces itself till about 20 mT below which the system suddenly goes superconducting. The data from a typical measurement is plotted in figure 3(a) for $V_g=200$ V. The superconducting state thus reached is transient and relaxes back to the original zero field resistive state with a time constant of around 10 seconds.
The appearance of this transient superconducting state depends critically on $dB/dt$, the rate at which the magnetic field is swept down from its maximum value. As shown in figure 4(a-c), for slow sweep rates of the magnetic field, there appears a dip in the resistance near 0 T, but the resistance remains finite. The magnitude of the dip increases as $dB/dt$ increases and beyond a certain value of $dB/dt$ the system goes into the transient superconducting state. The critical current beyond a certain value of $dB/dt$ the system goes into the transient superconducting state. The critical current is about 1 μA which matches well with the critical current measured in similar systems [12]. The appearance of the transient superconducting state depends also on the value of the highest magnetic field $B_{max}$ to which the system is taken before the field is ramped down. We observed that for $B_{max} < 6$ T, on reducing the field down to 0 T the system does not attain the transient superconducting state (see figure 3(c)). Interestingly, we also do not observe the transient superconducting state when the magnetic field is applied parallel to the interface [See Supplementary section].

To understand why the system changes from a ferromagnetic state to a magnetic field assisted transient superconducting state it is first necessary to understand the nature of the mobile charge carriers in the system. The dependence of sheet carrier number density $n$ on gate voltage is anomalous for $V_g > V_g^*$. In figure 5(a) we plot $n$ which we have extracted from the Hall measurement data assuming a single type of charge carrier in the system. We note that for $V_g > V_g^*$, $n$ appears to decrease with increase in $V_g$; simultaneously the Hall voltage $V_H$ develops a slight non-linearity with $B$. The charge carriers being electrons in this case, applying a positive gate voltage $V_g$ is expected to enhance the carrier density $n$, as can be seen from the plot of resistance vs $V_g$ in figure 5(b). In figure 5(c) we plot the estimated excess carrier $n_{calc}$ that would be induced in the system by the gate voltage-the estimate takes into account the electric field dependence of the dielectric constant of the STO substrate [14]. We find that $n_{calc}$ and $n$ match very well (to within a geometric factor) for $V_g > V_g^*$. For values of gate voltage beyond $V_g^*$, $n$ begins to drop below the expected range showing that the apparent decrease of $n$ with increasing gate voltage cannot be accounted for by the electric field dependence of the dielectric constant of the STO substrate.

**DISCUSSION**

The change of sign of the magnetoresistance from -ve to +ve around a certain value of $V_g$ has been observed before in LAO/STO heterostructures and has been interpreted to be due to a transition from weak localization (WL) to weak anti-localization (WAL) mediated by the large Rashba SOC present in this system [15]. To the best of our knowledge, a magnetic field assisted transient superconducting state has not been observed so far. In a related work a slight reduction in resistance on the insulating side of the superconductor-insulator transition was seen whose magnitude depended on $dB/dt$. This was interpreted as a signature of the presence of localized cooper pairs in the system in the non-superconducting state [16].

The fact that $n$ (as deduced from the slope of $V_H$) seemingly decreases with increase in $V_g$ beyond $V_g^*$ indicates that the transport in this regime is best described by a multi-band model [17]. It is known for LAO/STO heterostructures that at a certain number density, the system undergoes a Lifshitz transition between light and heavy sub-bands having different symmetries [18]. The additional carriers introduced are believed to occupy a higher mobility $d_{xz}/d_{yz}$ band near the interface and are responsible for the appearance of superconductivity in the system [17].

**Origin of the transient superconducting state:**

There exists now strong evidence, both experimental [19–21] and theoretical [22, 23], that superconductivity at the interface coexists with (in-plane) magnetization in phase segregated regions. The superconducting state is fragile with very low superfluid density [20, 21]. At low gate voltages our particular device is deep inside the ferromagnetic regime as seen from the large hysteresis in the magnetoresistance. The ferromagnetic nature is gradually weakened with increasing $n$ as evidenced by the gradual decrease and ultimately vanishing of hysteresis in MR near $V_g^*$. Beyond a certain critical density the system is in a metastable state - the itinerant electrons in the $d_{xz}/d_{yz}$ orbitals favour a superconducting ground state while the in-plane magnetization [24], which originates from the localized magnetic moments at the interface, opposes superconductivity, suppressing superconducting $T_c$. On application of a perpendicular magnetic field, magnetization of the (in-plane) FM-aligned domains reduces and when the field is ramped down, it takes a finite time for the in-plane magnetization to come back to its initial value. This creates a time window when the superconducting state is the lower energy state facilitating the emergence of the transient superconducting state. Therefore, at 245 mK, superconductivity is a hidden order and is masked by the in-plane magnetization- appearing only when the in-plane magnetization is sufficiently low. The fact that no transient superconducting state is seen for magnetic field applied parallel to the interface supports this picture. To understand quantitatively the origin of the transient superconducting state, we have computed the three components of magnetization and the superconducting gap parameter at each instant of time, as shown in figure 6, when the magnetic field (applied perpendicular to the interface) is ramped linearly with time [See Supplementary section for detailed calcu-
lations]. The life-time of the transient superconductivity obtained from our calculations is about 12 seconds (for $B_{max} \approx 8$ T and $dB/dt = 1$ T/min) which is pretty close to the experimentally observed value of about 10 seconds. The critical maximum magnetic field (≈ 6 Tesla), below which the transient superconductivity does not appear, also comes out of the calculation as shown in figure 6(c). Besides, it is experimentally seen that while decreasing the magnetic field, superconductivity appears at a finite critical field of 20 mT. From the theoretical analysis also it is possible to obtain a critical magnetic field (shown in y-axis on the right in figure 6(c)).

To conclude, we report in this letter the observation of a novel transient superconducting state. The transient superconducting state appears when a relaxing normal magnetic field reduces the in-plane magnetization to a value such that electron pairing becomes energetically favourable. This shows the inherently metastable nature of the superconducting state competing with a magnetic order. The coexistence of superconductivity and magnetic order and their controlled tunability using external field open up a new regime of investigation with tremendous potential in device applications.

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METHODS

Our measurements were performed on samples with 10 unit cells of LaAlO$_3$ grown by Pulsed Laser Deposition (PLD) on TiO$_2$ terminated (001) SrTiO$_3$ single crystal substrates. As received SrTiO$_3$ substrates were pre-treated with standard buffer HF solution [25] in order to achieve uniform TiO$_2$ termination. Prior to deposition the treated substrates were annealed for an hour at 830°C in oxygen partial pressure of $7.4 \times 10^{-2}$ mbar. Further, 10 unit cells LaAlO$_3$ was deposited at 800°C in the oxygen partial pressure of 1x $10^{-4}$ mbar. Growth with the precision of single unit cell is monitored by the oscillations count using in-situ RHEED gun. Magnetoresistance was measured using standard low frequency ac measurement technique in the temperature range 10 mK to 500 mK and up to 8 T magnetic field. The carrier density $n$ was tuned using a global back gate.

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Relaxation of the magnetoresistance: Figure S1 shows a plot of the relaxation of the sheet resistance as a function of time at 0.45 T magnetic field. To obtain this data, the magnetic field was initially ramped up from 0 T to 0.45 T at the rate 1 T/min. The magnet was then held constant at 0.45 T and the sheet resistance $R_{\text{sheet}}$ monitored as a function of time. It was seen that $R_{\text{sheet}}$ relaxes to a higher value over a couple of minutes. In a separate experiment, the magnetic field was ramped down starting from 8 T at the rate 1 T/min to 0.45 T, the field was held at 0.45 T and the resistance monitored as a function of time. It can be seen that in both cases the sheet resistance relax to the same value, although with slightly different time constants. Figure S2 shows the temperature evolution of the magnetoresistance. The hysteresis in MR weakens as the temperature is increased and eventually dies out by 2K.

Magnetoresistance in parallel field: Figure S3 shows the magnetoresistance with the magnetic field applied parallel to the plane of the two dimensional electron gas. In both configurations, with the field applied parallel to and perpendicular to the direction of the current, we do not observe any transient superconductivity.

Dynamics of magnetization and superconductivity: We start with a situation where the in-plane magnetization (taken along the x-axis) has completely destroyed the superconducting order. While increasing magnetic field, the dynamics of the three components of magnetization is described by the following set of Bloch’s equations:

$$\frac{dm_x}{dt} = \gamma B_z(t)m_y - \frac{m_x}{T_2}$$
$$\frac{dm_y}{dt} = -\gamma B_z(t)m_x - \frac{m_y}{T_2}$$
$$\frac{dm_z}{dt} = -\frac{m_z - m_{zs}}{T_1}$$

where $\gamma$ is called the Gyromagnetic ratio, $T_1$ and $T_2$ are the time-scales for the spin-lattice and spin-spin relaxation respectively. $B_z(t)$ is increased at the rate $dB/dt$ so as to reach the final value $B_{max}$. With the initial conditions $m_x(t=0) = m_{x0}$, $m_y(t=0) = 0$, $m_z(t=0) = 0$, the solutions to the above equations are

$$m_x(t) = m_{x0}\cos(\gamma B_z(t))e^{-t/T_2}$$
$$m_y(t) = -m_{x0}\sin(\gamma B_z(t))e^{-t/T_2}$$
$$m_z(t) = m_{zs}[1 - e^{-t/T_1}]$$

Therefore, the in-plane magnetization $m_x$ decreases exponentially from its initial value $m_{x0}$ while the out-of-plane magnetization $m_z$ grows up to its saturation value $m_{zs}$. Even though the magnetization $m_y$ along y-direction was zero initially, it attains a finite value and oscillates over a large range further degrading the electron pairing. Since perpendicular magnetization is much more detrimental to superconductivity than an in-plane one, it is not possible for the superconductivity to appear in this case.

When the magnetic field is decreased at the rate $dB/dt$ from the value $B_{max}$ at which the final magnetizations are $\{m_{xf}, m_{yf}, m_{zf}\}$, the set of equations describing the dynamics is:

$$\frac{dm_x}{dt} = \gamma B_z(t)m_y - \frac{m_x}{T_2}$$
$$\frac{dm_y}{dt} = -\gamma B_z(t)m_x - \frac{m_y}{T_2}$$
$$\frac{dm_z}{dt} = -\frac{m_z}{T_1}$$

The solutions of the above equation, with the initial conditions $m_x(t=0) = m_{xf}$, $m_y(t=0) = m_{yf}$, $m_z(t=0) = m_{zf}$, are

$$m_x(t) = [m_{xf}\cos(\gamma B_z(t)) + m_{yf}\sin(\gamma B_z(t))]e^{-t/T_2}$$
$$m_y(t) = [m_{yf}\cos(\gamma B_z(t)) - m_{xf}\sin(\gamma B_z(t))]e^{-t/T_2}$$
$$m_z(t) = m_{zf}[1 - e^{-t/T_1}]$$

While decreasing magnetic field, the localized moments at the interface start establishing the in-plane magnetization again to its initial value $m_{z0}$ according to

$$m_{z1}(t) = m_{z0}[1 - e^{-t/T_3}] + m_{zf}e^{-t/T_3}$$

which accompanies $m_x(t)$ in above equation. Therefore, $m_z$ starts decreasing from its saturation value $m_{zs}$ while $m_x$ starts increasing from a very low value back to its initial value. Since the relaxation time for $m_z$ to achieve its initial value is quite large, there exists a finite time slice in which the magnetizations along all directions are too weak to suppress the superconductivity. This will show up as the transient superconductivity in that narrow window of time with a $T_c$ higher than 200 mK. As
soon as $m_x$ grows to a larger value, the superconductivity is again destroyed. This relaxation of the in-plane magnetization causes the destruction of the already weak superconducting state. As shown in figure S1, the relaxation time during decreasing magnetic field is larger than that during increasing magnetic field. Therefore, in the calculation, the relaxation time constant $T_3$ has been taken large compared to $T_1$ or $T_2$ because the response of the spins to the magnetic field is much faster than to the growing magnetic moments. The theoretical estimate of critical B field (at which superconductivity appears while decreasing the field) is almost an order of magnitude larger than the experimentally observed value (at 6T) and could well come from strong dynamical disorder effects not included in the theory.

Model Hamiltonian and BdG treatment: We consider the following tight-binding Hamiltonian, to describe pairing in the interface electrons

$$H = -t' \sum_{<ij>,\sigma} (c_{i\sigma} \dagger c_{j\sigma} \dagger + \text{h.c.}) - \mu \sum_{i,\sigma} c_{i\sigma} \dagger c_{i\sigma} - \mu_B \sum_{i,\sigma,\sigma'} (h,\sigma)_{\sigma,\sigma'} c_{i\sigma} \dagger c_{i\sigma'} - i\frac{\alpha}{2} \sum_{<ij>,\sigma,\sigma'} (c_{i\sigma} \dagger \vec{\sigma}_{\sigma,\sigma'} \times \vec{d}_{ij}) \varepsilon c_{j\sigma},$$

$$+ \sum_{i} \Delta_i (c_{i\uparrow} \dagger c_{i\downarrow} \dagger + \text{h.c.})$$

where $t'$ is the kinetic hopping amplitude of electrons, $\mu$ is the chemical potential, $\mu_B$ is the Bohr magneton, $h = (m_x, m_y, m_z)$ represents the exchange fields due to the different components of magnetization, $\alpha$ is the strength of Rashba spin-orbit interaction, $\vec{d}_{ij}$ is unit vector between sites $i$ and $j$, and $\Delta_i = -U < c_{i\uparrow} c_{i\downarrow} >$ is the onsite pairing amplitude with the attractive pair-potential $U$.

The above Hamiltonian is diagonalized via a spin-generalized Bogoliubov-Valatin transformation $\hat{c}_{i\sigma}(\vec{r}_i) = \sum_{i,\sigma} u_{n\sigma\sigma'}(\vec{r}_i) \hat{\gamma}_{n\sigma} + v_{n\sigma\sigma'}(\vec{r}_i) \hat{\gamma}_{n\sigma'}$ and the quasiparticle amplitudes $u_{n\sigma}(\vec{r}_i)$ and $v_{n\sigma}(\vec{r}_i)$ are determined by solving the BdG equations

$$H \phi_n(\vec{r}_i) = \epsilon_n \phi_n(\vec{r}_i),$$

where, $\phi_n(\vec{r}_i) = [u_{n,\uparrow}(\vec{r}_i), u_{n,\downarrow}(\vec{r}_i), v_{n,\uparrow}(\vec{r}_i), v_{n,\downarrow}(\vec{r}_i)]$. The local pairing gap $\Delta_i$ is, therefore, obtained using the following relation:

$$\Delta_i = -U \sum_n [u_{n,\uparrow}(\vec{r}_i) v_{n,\downarrow}(\vec{r}_i) (1 - f(\epsilon_n))$$

$$+ u_{n,\downarrow}(\vec{r}_i) v_{n,\uparrow}(\vec{r}_i) f(\epsilon_n)]$$

where, $f(x) = 1/(1 + \exp(x/k_B T))$ is the Fermi function at temperature $T$ with $k_B$, the Boltzmann constant. At any instant of time $t$, the components of magnetization are calculated and then inserted into the above Hamiltonian to solve the mean-field pairing gap self-consistently.
Figure 1. (a) Resistance of the device as a function of temperature at different gate voltages ranging from -25V till 200V. (b) Plot of the sheet resistance $R_{\text{sheet}}$ as a function of temperature $T$ and gate voltage $V_g$ showing the superconducting dome.

Figure 2. (a) Magnetoresistance (MR) of the device at different gate voltages ranging from -200V till 170V. The measurements were all done with the device temperature at 245 mK. (b) Magnetoresistance at gate voltage $V_g = -200V$ showing hysteresis at low magnetic fields. (c) Hysteresis in magnetoresistance as a function of gate voltage and magnetic field at 245 mK. Note that the hysteresis gradually dies out as the gate voltage increases.
Figure 3.  (a) Magnetoresistance at gate voltage $V_g = 170$V and $T = 245$ mK showing the transient superconducting state. In this measurement the magnetic field was swept down at a rate $dB/dT = 1$ T/min.  (b) Time relaxation of the transient superconducting state at two different gate voltages measured after the magnetic field has been swept down to 0 T at a rate $dB/dT = 1$T/min. The system goes from the zero resistance state thus achieved to the value at normal zero magnetic field over a time period of a few tens of seconds. (c) Effect of the maximum field on the transient superconducting state. Note that for $B_{\text{max}} = 5$ T the transient superconducting state is not reached. The measurement was done at $V_g = 135$ V and $T = 245$ mK.

Figure 4. Effect of different sweep rates of the magnetic field on the transient superconducting state - (a) $dB/dt = 0.1$ T/min, (b) $dB/dt = 0.2$ T/min and (c) $dB/dt = 0.5$ T/min. The measurements were done at $V_g = 110$ V and $T = 245$ mK.
Figure 5. (a) Number density calculated from Hall measurements as a function of the $V_g$ and (b) zero magnetic field sheet resistance as a function of the $V_g$; measured at a temperature 245 mK. The colours of the filled circles correspond to the colour of the resistivity curve at zero magnetic field plotted in figure 1 (a) and the magnetoresistance curves plotted in figure 2 (a). (c) The coloured filled circles are the measured excess charge induced in the system due to the gate voltage, the solid line is the expected value taking into account the electric field dependence of the dielectric constant of SrTiO$_{3}$.

Figure 6. Time-variation of the magnetic field, the three components of magnetization and the mean superconducting gap while (a) increasing and (b) decreasing the magnetic field (with maximum field strength $B_{\text{max}} = 7$ T). (c) Variation of the life-time of superconductivity and the critical B-field, as $B_{\text{max}}$ is varied. Parameters: $t' = 0.277 eV$, $\mu = 0$, $\alpha = 20 meV$, $U = t' eV$, $dB/dt = 1T/min$, $B_{\text{max}} = 7T$, $\gamma = 1$, $T_1$ (B increasing) = 200 sec, $T_2$ (B increasing) = 100 sec, $T_1$ (B decreasing) = 400 sec, $T_2$ (B decreasing) = 200 sec and $T_3 = 550$ sec.
Figure S1. (a) Relaxation of the magnetoresistance with time. The magnetic field was initially ramped up from 0 T to 0.45 T at the rate 1 T/min. The field was then held at 0.45 T and the sheet resistance $R_{\text{sheet}}$ monitored as a function of time. In a separate experiment, the magnetic field was ramped down starting from 8 T at the rate 1 T/min to 0.45 T, the field was held at 0.45 T and the resistance monitored as a function of time. The measurements were done at $V_g = -200$ V and temperature 245 mK. (b) Plot as a function of $V_g$ of the magnetic field $B_{\text{corr}}$ at which the hysteresis in perpendicular magnetoresistance is maximum. Note that as $V_g$ increases $B_{\text{corr}}$ decreases and ultimately vanishes for $V_g > 40$ V.

Figure S2. Magnetoresistance as a function of temperature at $V_g = -200$V. (a) Sheet resistance as a function of magnetic field at different temperatures. (b) Plot of the hysteresis in magnetoresistance as a function of magnetic field at different temperatures - the hysteresis is seen to decrease sharply as the temperature increases.
Figure S3. Magnetoresistance of the device at different directions of the magnetic field with respect to the device at $T = 245$ mK and (a) $V_g = -200V$, (b) $V_g = 110V$. In the longitudinal configuration the magnetic field was parallel to the conducting layer at the interface and also to the direction of the current. In the tranverse configuration the magnetic field was parallel to the conducting layer at the interface and perpendicular to the direction of the current. The transient superconducting state only appears for $V_g > V_g^*$ when the magnetic field direction is perpendicular to the conducting layer at the interface.