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Fabricating superconducting interfaces between artificially grown LaAlO$_3$ and SrTiO$_3$ thin films

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Realization of a fully metallic two-dimensional electron gas (2DEG) at the interface between artificially grown LaAlO$_3$ and SrTiO$_3$ thin films has been an exciting challenge. Here we present for the first time the successful realization of a superconducting 2DEG at interfaces between artificially grown LaAlO$_3$ and SrTiO$_3$ thin films. Our results highlight the importance of two factors—the growth temperature and the SrTiO$_3$ termination. We use local friction force microscopy and transport measurements to determine that in normal growth conditions the absence of a robust metallic state at low temperature in the artificially grown LaAlO$_3$/SrTiO$_3$ interface is due to the nanoscale SrO segregation occurring on the SrTiO$_3$ film surface during the growth and the associated defects in the SrTiO$_3$ film. By adopting an extremely high SrTiO$_3$ growth temperature, we demonstrate a way to realize metallic, down to the lowest temperature, and superconducting 2DEG at interfaces between LaAlO$_3$ layers and artificially grown SrTiO$_3$ thin films. This study paves the way to the realization of functional LaAlO$_3$/SrTiO$_3$ superlattices and/or artificial LaAlO$_3$/SrTiO$_3$ interfaces on other substrates.

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approach also opens possibilities for exploiting the strain effect, which in the LAO/STO interface still awaits full exploration.\textsuperscript{17} More interestingly, the capability to build the LAO/STO interface on artificially grown STO is also fundamental for realizing LAO/STO superlattices. In fact, a few reports have discussed different physical aspects of LAO/STO superlattices,\textsuperscript{18,19} whereas a clear experimental demonstration of multiple parallel conducting 2DEGs is still elusive. Such heterostructures would allow, for instance, the study of the coupling between 2D superconductors.\textsuperscript{20} In fact, the major challenge to achieve these goals is to obtain a high quality metallic interface on an artificially grown STO film. To date, metallic two-dimensional interfaces down to low temperatures have only been observed when LaAlO\textsubscript{3} is grown directly on single-crystalline SrTiO\textsubscript{3} substrates. Efforts have been made towards realizing such interfaces between LAO and artificially grown STO films and conducting LAO/STO interfaces grown on other substrates have been achieved.\textsuperscript{21–24} However, the metallic behavior was no longer maintained in the low temperature range and localization effects were observed.\textsuperscript{23} The main obstacle remains to be preserving both the metallicity down to the lowest temperature and the two-dimensional nature of the electron gases by using proper oxygen deposition conditions.\textsuperscript{23}

In this article we explore the fabrication of an artificial LAO/STO interface, where the 2DEG is formed within a STO layer that was homoepitaxially grown on a (001) STO single-crystal. We demonstrate that the 2DEG at the fabricated interfaces is not only metallic down to the lowest temperatures, but also superconducting. Analysis of the transport properties shows that the growth temperature determines the crystalline perfection of the STO layer and consequently the metallicity of the system. Using Friction Force Microscopy (FFM), X-ray diffraction, and transport measurements, we relate the absence of metallic behavior normally observed in LAO/STO thin films to the locally nanoscale SrO segregation on the STO surface and to the associated defects in the STO thin films. Our findings show that by controlling the top surface termination of the STO layer and optimizing the STO growth, the 2DEG can be preserved, offering a promising approach to realizing functional LAO/STO multilayers and/or superconducting LAO/STO interfaces on other substrates.

LAO and STO films were grown by pulsed-laser deposition (PLD) from single-crystalline targets. The laser fluence was set to $\sim 0.6$ J cm$^{-2}$ and the repetition rate was kept at 1 Hz. The growth was monitored by reflection high-energy electron diffraction (RHEED). We followed the evolution of the intensity of the specular spot to identify the growth mode. The STO films were grown on TiO\textsubscript{2}-terminated (001)-oriented STO single-crystalline substrates at 800°C in an oxygen pressure of $8 \times 10^{-5}$ Torr and at 1100°C in an oxygen pressure of $1 \times 10^{-6}$ Torr. The LAO layers were grown at 800°C in an oxygen pressure of $8 \times 10^{-5}$ Torr. The samples characterized in this work are classified into two categories, depending on the fabrication process. For convenience, we name them \textit{in situ} samples and \textit{ex situ} samples. As shown in Figure 1, for \textit{in situ} samples, a STO thin film is grown on a (001) TiO\textsubscript{2}-terminated STO substrate, followed by the deposition of a LAO film without breaking the vacuum. For the \textit{ex situ} samples, the following fabrication steps were employed: (a) STO film deposition on a (001) TiO\textsubscript{2}-terminated STO substrate; (b) \textit{ex situ} surface treatment of the STO layer (buffered-HF etching followed by a high-temperature annealing procedure);\textsuperscript{25,26} and (c) LAO film deposition. Extra care was taken to avoid as much as possible the formation of oxygen vacancies, both in the STO films and in the substrates, by performing an oxygen-annealing step during sample cooling in the deposition chamber. In this manner, we fabricated \textit{in situ} and \textit{ex situ} samples with STO thin films of different thicknesses (from 2 u.c. to 40 u.c.) grown at different temperatures (800°C and 1100°C), while keeping the LAO thickness constant (5 u.c.). All the scanning probe measurements were performed with a Cypher microscope (Asylum Research) in contact mode. Sb-doped Si cantilevers with a nominal force constant of $k = 0.2$ N m$^{-1}$ were used. The friction contrast was then obtained by a subtraction of the lateral retrace signal from the lateral trace one. No attempts have been made to quantify the absolute friction force since the lateral force constant of the cantilever is unknown. The magneto-transport measurements were performed in a He\textsubscript{4} cryostat equipped with a superconducting magnet (8 T). Field-effect devices were prepared by depositing a gold pad as the gate electrode on the backside of the STO substrate. The superconducting transitions were measured in a He\textsubscript{3}/He\textsubscript{4} dilution refrigerator with a base temperature of 30 mK.

We first focus on the \textit{in situ} and \textit{ex situ} samples with STO layers grown at 800°C. At this substrate temperature, STO grows layer by layer, as revealed by periodic RHEED intensity oscillations.\textsuperscript{27}
Figure 2(a) displays a sketch of a sample patterned into Hall-bar geometry for transport measurements. Figure 2(b) shows the room-temperature sheet conductance as a function of the STO layer thickness ($d$, in u.c.), for both the in situ and ex situ samples. From the figure, one can clearly see that for in situ samples above $d = 8$ u.c. the system begins to lose conductivity: the sheet conductance falls outside the range of standard LAO/STO interfaces indicated by the dashed area, and drops below the measurement limit for $d = 15$ u.c. For ex situ samples, the $d = 15$ u.c. sample is still metallic and thus the critical STO thickness for metallicity shifts upwards. This shift is also reflected in the sheet carrier density estimated from Hall effect measurements. Figure 2(c) shows $-1/eR_H$ ($R_H$ being the Hall constant) for the same series of samples. We note that, for the ex situ samples, there is a clear tendency towards a carrier density reduction before they become immeasurable as the STO layer thickness is increased.\(^{27}\) The comparison between in situ and ex situ samples highlights the importance of the surface treatment, suggesting that the TiO$_2$ termination may not be preserved during the growth of the STO films at 800°C.\(^{28}\) In this case, regions with SrO termination could reduce the global conductivity of the system as evidenced by previous work where the deposition of a SrO layer on the STO surface was shown to inhibit the formation of the 2DEG at the interface.\(^{5, 26, 29}\)

To investigate the possible presence of SrO on the surface, we used the friction force mode (FFM) of an atomic force microscope. FFM probes the nanoscale friction force as the tip is dragged across the scanned surface by monitoring the cantilever torsion during the scan. By subtracting the retrace scan from the trace scan of the lateral signal, one can disentangle the contribution due to the different friction coefficients from the one purely related to the topography, such as unit-cell step edges, thus obtaining a clear contrast with nanoscale resolution linked to the local terminations.\(^{27}\) This approach has been previously employed to determine the local termination of complex-oxide surfaces and in particular to probe the partial SrO coverage on the surface of STO single-crystals.\(^{30, 31}\)

Figures 3(a) and 3(b) show the surface topography and the FFM images, respectively, of a single-crystalline (001) STO substrate. Unit-cell-high step-and-terrace structure, a characteristic feature of a miscut TiO$_2$-terminated STO surface, can be clearly seen on the topographic image. Despite these unit-cell steps, no contrast is visible on the friction image, suggesting uniform TiO$_2$ coverage. On top of such substrates, we grew 15 u.c. thick STO films, since for this layer thickness the in situ and ex situ LAO/STO/STO structures showed fundamentally different transport properties (insulating and metallic, respectively). After the STO deposition, the surface becomes rougher, marked by the presence of nanometer-sized pits, islands, and rough step edges, as shown in Figures 3(c) and 3(e). This may already indicate that the TiO$_2$ termination is no longer well maintained and mixed termination appears. However, this feature is not resolved in the friction images shown in Figures 3(d) and 3(f), where no clear contrast is distinguishable. This may be due to the
FIG. 2. Transport properties of the LAO/STO/STO samples with STO layers grown at 800 °C. (a) A sketch of the LAO/STO/STO sample with a Hall-bar pattern used to measure the sheet resistance and carrier density of the system. (b) Sheet conductance at 280 K; (c) $-1/e R_H$ at 5 K versus STO layer thickness $d$ (u.c.). Black open (blue solid) circles are for in situ (ex situ) samples. The shaded areas in (b) and (c) indicate the room-temperature sheet conductance and $-1/e R_H$ at 5 K, respectively, in standard LAO/STO interfaces. The lines are guides to the eye.

fact that the termination of the surface is mixed on sub-nanometer scale that is below the resolution of FFM.

In order to detect the presence of SrO, we performed an annealing treatment in an oxygen atmosphere at ambient pressure on one 15 u.c. thick STO film. Such an annealing promotes the migration of surface species to the step edges, and is routinely used as a complementary step after the buffered HF (BHF) etching to obtain the atomically flat TiO$_2$ termination with sharp unit-cell step edges. Since a long high-temperature anneal may also promote SrO segregation on the STO surface, we keep our annealing time below 2 h. Figures 3(g) and 3(h) illustrate the topographic and friction images after the annealing treatment. In the friction scan, we observe the appearance of a clear contrast, with nanometer-sized white areas present at the terrace edges that we relate to the accumulation of SrO. In order to exclude the possible segregation of SrO during the annealing step, we performed a control experiment by treating an identical 15 u.c. thick STO film first with BHF and then with the same annealing process. The topography and friction images illustrated in Figures 3(i) and 3(j) do not suggest any presence of SrO as we do not observe any contrast in the FFM scan. Therefore, using these treatment conditions, once the segregated SrO is removed by the BHF etching, the TiO$_2$ termination is preserved during the high-temperature annealing.

We can then conclude that the SrO segregation occurs on the film surface during STO growth and may be responsible for the loss of the 2DEG at the interface for in situ samples. We should also note that this loss of the 2DEG properly highlights the fact that oxygen vacancies, which can possibly be formed during the growth, seem not to be the relevant origin of the metallicity in this study. Looking at Figures 2(b) and 2(c), however, raises the question: why is this loss also observed for ex situ samples, albeit at higher STO critical thickness?
To address this question, we have analyzed the structural quality of the STO films grown at 800 °C. Figure 4(a) shows a θ-2θ X-ray diffractogram around the (002) Bragg reflection of STO. Around the substrate peak we observe oscillations attributed to the finite thickness of the layer. A fit to these oscillations yields a film thickness of 40 u.c., in agreement with the estimation from the count of the RHEED oscillations. The presence of these oscillations suggests a slight off-stoichiometry of the STO film, probably originating from the SrO migration and segregation to the surface. These defects in the STO films could induce localization of the 2DEG, resulting in an insulating interface. In order to improve the quality of the STO films, we have raised the growth temperature to 1100 °C. Previous work has shown that above 1000 °C, STO films grow by step-flow and have dielectric properties comparable to those in single crystals. Indeed, during the deposition, we observe that the RHEED specular intensity recovers completely after each laser pulse, indicating step-flow growth. X-ray diffraction data for samples grown at high temperature reveal only the substrate peaks with no finite size oscillations. In Figure 4(a) we compare a θ-2θ scan around the (002) reflection for the 800 °C sample and for the 1100 °C sample, both being 40 u.c. thick. The absence of the finite-size oscillations indicates that the film is indistinguishable from the single crystalline substrate. The AFM investigation of the 40 u.c. film surface before and after a thermal treatment (but without BHF etching) is summarized in Figures 4(c) and 4(d), and 4(e) and 4(f), respectively. The topography images show atomically flat surface while no friction contrast is observed in the FFM images, indicating the absence of SrO segregation for these films.

We prepared a series of ex situ samples with STO layers of different thickness grown at 1100 °C. In Figure 4(b), the room-temperature sheet conductance of these samples is compared with that of ex situ samples grown at 800 °C. One can see that for STO layers grown at 1100 °C, the 2DEG is preserved for STO films with a thickness of 40 u.c. or about 16 nm (the maximum thickness we used). This value exceeds the spatial extent of the 2DEG, which has been shown to be 1–2 u.c. at room temperature and a few nanometers at low temperature. All the samples with STO layers grown at 1100 °C show metallic behavior down to the lowest temperature (1.5 K). Finally, we also fabricated one in situ sample with a STO thickness of 40 u.c., which also displays the fully metallic behavior and whose room-temperature sheet conductance is also indicated in Figure 4(b). This suggests that increasing the STO growth temperature not only preserves the STO crystal quality but also its TiO2 surface termination.

Figure 5 shows a detailed characterization of the transport properties of ex situ LAO/STO samples with a 40 u.c. thick STO layer grown at 1100 °C. The values of the carrier density (estimated...
FIG. 4. (a) X-ray diffraction patterns of 40 u.c. homoepitaxial STO films grown at 800 °C and 1100 °C. The arrows indicate finite size oscillations. (b) Sheet conductance at 280 K for STO layers of different thickness $d$ (u.c.). Blue circles (black squares) are for ex situ samples with STO layers grown at 800 °C (1100 °C). The red star is for an in situ sample with the STO layer grown at 1100 °C. The shaded area indicates the room-temperature sheet conductance in standard LAO/STO interfaces. The lines are guides to the eye. (c) VFM and (d) FFM images of a 40 u.c. thick STO film grown on a TiO2-terminated STO substrate at 1100 °C. (e) VFM and (f) FFM images of the same sample after annealing at 1000 °C.

via the Hall effect) and of the electron mobility shown in Figures 5(a) and 5(b) are in line with standard LAO/STO interfaces.\textsuperscript{42} In particular, we do not observe a suppression of the carrier mobility and a strong localization behavior, as reported in Ref. 23. However, we also do not observe an enhancement of the carrier mobility. In order to further characterize this artificial interface, field-effect experiments in back-gate geometry were performed; in this configuration, the STO layer and the STO substrate act as the series gate dielectrics. The large tunability of the sheet resistance and the evolution of the magneto-resistance with gate voltage shown in Figure 5(c) are very similar to those of standard LAO/STO interfaces. These results confirm that high temperature growth enables the fabrication of high quality interfaces. Superconductivity was also observed at these interfaces made of artificially grown LAO and STO thin films in the millikelvin temperature range. Figure 5(d) illustrates the normalized resistance versus temperature for a standard LAO/STO interface and for a sample with a 40 u.c. thick STO layer grown at 1100 °C. A clear and complete superconducting transition is observed for the STO thin film sample albeit with a lower $T_c$ of about 100 mK. This can be related to a different doping state and may suggest differences in the superconducting state that need to be fully explored. Given that the thickness of the superconducting electron gas at LAO/STO interfaces is $\sim$10 nm,\textsuperscript{41} these results demonstrate, for the first time, the successful generation of a two-dimensional superconducting electron gas between artificially grown LAO and STO thin films. These results open a new pathway to realize the LAO/STO superlattices with multiple parallel 2D superconductors.

In conclusion, we have presented a successful realization of a superconducting 2DEG at interfaces between artificially grown LAO and STO thin films. Our results highlight the importance of two factors—the growth temperature and the STO termination. Samples grown at 800 °C do not host the 2DEG for STO layer thicknesses above a critical value. The observation of nanoscale SrO segregation on the STO surface by the local friction force microscopy and the presence of defects in the STO layer seems to be plausible origins for this behavior. Increasing the deposition temperature for the STO layers to 1100 °C changes the growth mode to step-flow and produces
films of higher quality; a metallic, down to the lowest temperatures, and superconducting 2DEG is observed on these artificial LAO/STO interfaces even for thick STO layers. This study provides the key ingredients needed to grow LAO/STO superlattices or interfaces on other substrates, where exciting physical properties can be realized and studied.

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