Spin–orbit interaction and Kondo scattering at the PrAlO$_3$/SrTiO$_3$ interface: effects of oxygen content

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Received 24 April 2017, revised 3 July 2017
Accepted for publication 12 July 2017
Published 23 August 2017

Abstract
We report the effects of oxygen pressure during growth ($P_{O_2}$) on the electronic and magnetic properties of PrAlO$_3$ films grown on TiO$_2$-terminated SrTiO$_3$ substrates. Resistivity measurements show an increase in the sheet resistance as $P_{O_2}$ is increased. The saturation of the sheet resistance down to 0.3 K is consistent with Kondo theory for $P_{O_2} \geq 10^{-5}$ torr. Resistivity data fits indicate Kondo temperatures of 16–18 K. For the $10^{-4}$ sample, we measured a moderate positive magnetoresistance (MR) due to a strong spin–orbit (SO) interaction at low magnetic fields that evolves into a larger negative MR at high fields due to the Kondo effect. Analysis of the MR data permitted the extraction of the SO interaction critical field for the $P_{O_2} = 10^{-5}$ torr interface ($H_{SO} = 1.25$ T). We observed high positive MR for the least oxygenated sample, where a fraction of the $n$-type carriers are derived from oxygen vacancies and possible cation interdiffusion; for this $6 \times 10^{-6}$ torr sample, Hall effect data indicate a thick conducting layer. Its extremely high MR ($\sim 400\%$) is attributed to classical behavior due to a distribution of mobilities.

Keywords: two-dimensional electron gas, interface, PrAlO$_3$/SrTiO$_3$, spin–orbit interaction, Kondo effect, high magnetoresistance

(Some figures may appear in colour only in the online journal)
mechanisms. The reason for cation mixing is thought to be the reduction in the dipole energy at the interface, and it has been shown that $La_{1-x}SrTiO_3 (0.05 < x < 0.95)$ is metallic [14, 16]. It was also experimentally [17, 18] and theoretically [19, 20] demonstrated that varying oxygen pressure during growth results in dramatic changes in interfacial conductivity. Moreover, conductivity found at the interface of STO and amorphous LAO [21, 22] suggests that oxygen vacancies can be a dominant source of mobile carriers. Oxygen post-annealing of the amorphous samples fills the oxygen vacancies, and the interface becomes insulating.

Strong spin–orbit (SO) coupling effects whose magnitude can be modulated by the application of an external electric field have been observed for the LAO/STO interface through magnetotransport studies [23]. This SO coupling effect that can be modulated by the application of an external electric field have been observed for the LAO/STO interface through magnetotransport studies [23]. This SO coupling effect that can be a dominant source of mobile carriers. Oxygen post-annealing of the amorphous samples fills the oxygen vacancies, and the interface becomes insulating.

Similar interfaces such as the interface of STO and PrAlO$_3$ [26], NdAlO$_3$ [27], LaGaO$_3$ [28], NdGaO$_3$ [29] and LaTiO$_3$ [30], where the film is polar, have all shown conductivity. We examined, in detail, the effects of oxygen content on the electrical and magnetic properties of PrAlO$_3$ (PAO) and STO for two main reasons. First, among the aluminates, PAO has the closest lattice constant to LAO (due to the multivalency of Ce, it is hard to make stable thin films of CeAlO$_3$), and thus this helps to lessen the effect of strain as much as possible. Second, the effective magnetic moment of Pr is 3.6 Bohr magnetons, due to Pr$^{3+}/f$ electron states, often hybridized, while closed shell La$^{3+}$ is diamagnetic [31], thus this rare-earth substitution can lead to magnetic properties in an aluminate structure. At room temperature bulk PAO is rhombohedral $R3\bar{c}$ with in-plane lattice parameters of 3.77 Å [32]. PAO is known to be paramagnetic in bulk and has mostly been studied for its structural transitions below room temperature which are absent in LAO [26-11].

This paper is organized as follows. Section 2 contains our experimental method for synthesizing and characterizing samples. Section 3 contains our experimental results of resistance and Hall effect as a function of temperature and magnetoresistance as a function of magnetic field for samples that are made at different oxygen pressures during growth. Comparison with theoretical concepts are made. A summary and conclusions are given in section 4.

2. Experiments

Epitaxial thin films were grown in a custom-built Pulsed Laser Deposition (PLD) vacuum chamber using a Lambda Physik COMPeX 201KrF (248 nm) pulsed excimer laser. The TiO$_2$-terminated SrTiO$_3$ (001) substrate was prepared by etching the substrate in buffered hydrofluoric (HF) acid for one minute and then by thermal annealing at 950 °C for 2h. To keep the effect of the substrate defect constant, a single substrate was cut into equal pieces to be used for depositing the different samples. PAO epitaxial thin films were prepared in the oxygen partial pressure ($P_{O_2}$) range of $6 \times 10^{-6}$ to $6 \times 10^{-4}$ torr at 750 °C. The laser pulse energy was 100 mJ, the fluence of the laser was $\sim 3$ J/cm$^2$ and the repetition rate was 1 Hz. After deposition, the samples were post annealed for 30 min and then cooled down to room temperature, both in the same oxygen pressure as during deposition. The quality of the terminated substrate and the thin film samples were further confirmed by atomic force microscopy (AFM) using a Veeco Nanoscope, *ex situ* at room temperature using tapping mode. The crystal phase and epitaxial growth of the films were further confirmed by x-ray diffraction (XRD) with a Philips X’PERT diffractometer using CuK$_\alpha$ x-rays. Alignment was achieved using the (002) reflection of STO. The buried interface was contacted in a Van der Pauw geometry by ultrasonic welding using a West Bond Inc. model 7476D manual aluminum wedge–wedge wire-bonder. Transport properties were measured using a 9 T Quantum Design physical property measurement system (PPMS). The growth oxygen pressures reported were from an ion gauge in close proximity to the substrate, but should be considered approximate, since pressure gradients are inherent between oxygen source and vacuum pump.

3. Results and discussions

3.1. Crystal structure and surface analysis

Figure 1(a) shows an XRD scan of epitaxial PAO films on STO (001) that were grown at different $P_{O_2}$. The thin films were grown along the $c$ axis since only the (00$l$) reflections of the PAO are present. The inset shows an x-ray reflectivity (XRR) scan of the $1 \times 10^{-4}$ torr sample. From a simulation fit, the thickness is estimated to be 6.8 nm, or 18 unit cells (uc). Similar results were obtained for the other samples. There was no significant change in the structure, crystallinity, and quality of these samples as a function of oxygen pressure during growth. Figure 1(b) depicts the AFM topography of a PAO/STO sample grown at $1 \times 10^{-4}$ torr. The step terrace structure of the substrate is maintained, verifying the layer-by-layer step-flow growth.

3.2. Sheet resistance and Kondo effect

In figure 2 we present the dependence on $P_{O_2}$ of the sheet resistance $R_s$ of the interfaces of PAO and STO. We observe that the samples become less conductive for higher $P_{O_2}$. The room temperature resistivity increases as the samples are prepared at higher oxygen pressure. While there is only less than a factor of 2 in growth pressure difference between the $6 \times 10^{-6}$ torr and $1 \times 10^{-5}$ torr samples, their room temperature resistivities differ by three orders of magnitudes. In the case of the LAO/STO interface, it is known that oxygen vacancies can provide intragap donor levels close to the conduction band of STO [34]. Higher oxygen pressure during growth tends to fill the oxygen vacancies, thus causing the room temperature $R_s$. 

J. Phys.: Condens. Matter 29 (2017) 395002
to increase. The lowest oxygen-pressure-grown sample shows a metallic behavior from room temperature down to 2 K. The resistivity for this sample levels off below 10 K. The very low sheet resistance and high carrier density (below) indicate that the conduction layer is much thicker for the high pressures higher than $10^{-4}$ torr. We first extended the measurement to 0.3 K for the high $P_{O_2}$ sample. To study the upturn in resistivity in more detail, we extended the measurement to 0.3 K for the range from 100 K to 10 K. The upturn is substantially stronger for the samples thicker than $10^{-4}$ torr. These variations in $R_s$ suggest that different types of transport mechanisms are operating at the interface. Some of the scattering processes that result in an upturn in resistivity are: scattering from localized magnetic moments (Kondo effect), weak localization (WL), and variable range hopping due to strong localization. The presence of a saturating resistance below $T_{K}$ is the signature feature of the Kondo effect and our data are best fit to a Kondo model. In the Kondo model an impurity in a metal couples to reservoir of itinerant electrons by an antiferomagnetic exchange coupling forming a virtual bound state. This coupling can result in a contribution to the resistivity through a universal function of $R_K(T/T_K)$, in units of the Kondo temperature $T_K$.

Metallic resistance including a Kondo effect term is given by:

$$R^\text{M}(T) = R_0 + \alpha T^2 + \beta T^5 + R_K(T/T_K) .$$  \hspace{1cm} (1)

Here, $R_0$ is the residual resistance due to disorder and impurities, $\alpha T^2$ is the $e$–$e$ interaction term and $\beta T^5$ is the contribution from the electron–phonon interaction. $R_K(T/T_K)$ represents the contribution of magnetic ions to electrical resistivity. $R_K(T/T_K)$ has a logarithmic trend at $T \gg T_K$ and a saturating behavior at $T \ll T_K$.

To fit our experimental data we used the empirical form of $R_K(T/T_K)$ presented by Costi [36] and Goldhaber [37], and later used by Lee et al in [38] for undoped STO gated by an ionic gel electrolyte. They have demonstrated a Kondo effect, as a function of an applied electric field, which is the result of interaction between magnetic Ti$^{3+}$ ions, unpaired and localized, with delocalized electrons that partially fill the Ti 3 $d$ band. That empirical form is:

$$R_K \left( \frac{T}{T_K} \right) = R_K(T = 0) \left( \frac{T^2}{T^2 + T_K^2} \right)^s .$$  \hspace{1cm} (2)

Here, $T_K$ is defined as the temperature at which the Kondo resistivity is half of the value of resistivity at zero temperature, $R_K(T_K) = R_K(T = 0)/2$ so that $T_K = T_K/\sqrt{2^{1/2} - 1}$. $s$ is the effective spin of the magnetic scattering centers. We chose $s = 0.75$ for both the $10^{-5}$ and $10^{-4}$ torr samples. We first fit the model to the high temperature part of the curves and extracted $\alpha$, $\beta$ and $R_0$, and then used these values for fitting to low temperatures (below 10 K) to extract $T_K$. Table 1 summarizes the values obtained from the fitting to equations (1) and (2) across the whole measured temperature range.

From table 1, we see an increase of about 2 $K$ in the $T_K$ values as the $P_{O_2}$ is increased from $10^{-5}$ torr to $10^{-4}$ torr.
The temperature dependences of sheet carrier density \(n_s\) and mobility \(\mu\) are displayed in figures 3(a) and (b), respectively. The positive values for \(n_s\) correspond to an electron-doped interface. For the \(P_{O_2} = 6 \times 10^{-6}\) torr sample, the carrier concentration is two orders of magnitude larger than the amount predicted by the polar catastrophe \((3.2 \times 10^{14} \text{ cm}^{-2})\), if half an electron per unit cell is transferred to STO surface from LAO. The \(n_s\) values for the lowest \(P_{O_2}\) sample are so high that it resembles a bulk metallic sample. This implies that oxygen vacancies or interdiffusion are contributing to the number of donors and creating a thick conducting region for the \(P_{O_2} = 6 \times 10^{-5}\) torr sample.

Also, for this sample, the mobility monotonically increases by three orders of magnitude as the sample is cooled down from room temperature, figure 3(b). For other samples \(\mu\) drops by further cooling below 40 K, perhaps due to localization. The number of sheet carriers for the \(P_{O_2} = 10^{-4}\) torr sample is smaller than the \(10^{-5}\) torr sample. For these two samples the numbers suggest 2D conductivity. There are some variations in \(n_s\) and \(\mu\) at certain temperatures; those that happen at 150 K and 220 K are around the structural transitions of PAO in bulk. At room temperature, PrAlO\(_3\) has a rhombohedral structure which transforms to an orthorhombic structure upon lowering the temperature to about 205–225 K, and to a monoclinic structure at about 151–175 K [33, 39]. A distinct drop of \(n_s\) at 32 K has been previously seen for the LAO/STO [35] interface and for the NAO/STO interface [27] at around 20 K.

### Table 1. Parameters obtained from fitting sheet resistance data of figure 2 to equations (1) and (2).

| Parameter          | \(10^{-5}\) | \(10^{-4}\) |
|--------------------|-------------|-------------|
| \(s\)              | 0.75        | 0.75        |
| \(R_0\) (\(\Omega\)) | 2090        | 635         |
| \(\alpha\) (\(\Omega\ \text{K}^{-2}\)) | 0.153       | 0.39        |
| \(\beta\) (\(\Omega\ \text{K}^{-2}\)) | 4.5 \times 10^{-11} | 3.6 \times 10^{-9} |
| \(R_0(T = 0)\) (\(\Omega\)) | 3500       | 27000       |
| \(T_k\) (K)        | 16.1        | 18.3        |

3.3. Magnetoresistance and spin orbit interaction

Figure 4(a) shows the magnetic field dependence of the magnetoresistance (MR) at 2 K. MR data are calculated as MR = \([R(H) - R(0)]/R(0)\) \times 100\%. The measurement was done both with the magnetic field \(H\) perpendicular to the interface plane (out-of-plane MR), figure 4(a), and with the magnetic field \(H\) parallel to the interface plane (in-plane MR), figure 4(b). We removed the Hall voltage by measuring the resistivity at +/−\(H\) fields and then took the average of those two measurements. That eliminates the antisymmetric contribution from the Hall voltage, as the Hall voltage changes sign when the field changes direction.

For the \(6 \times 10^{-6}\) torr sample, (insets of figures 4(a) and (b)), MR values are positive for all measured fields and are immensely large (300–400 % at 9 T); the field dependence resembles some bulk metals. Very high positive MR has also recently been reported in degenerate semiconductive STO single crystals capped with ultra thin STO/LAO bilayers [40]. The transport model of Parish and Littlewood [41, 42] attributes similar extremely high MR values to a distribution of local mobilities due to disorder, appropriate for this sample with appreciable oxygen vacancies and interdiffusion.

For the \(P_{O_2} = 10^{-5}\) torr interface, the MR data has a positive slope at low fields and a negative slope at high fields, resulting in a local maximum in the MR.

We attribute the positive part of the MR to strong spin–orbit (SO) scattering and the negative part to the Kondo mechanism. The SO interaction has been shown to be very strong in some of the semiconductor heterostructures such as \(p\)-type GaAs/Al\(_{x}\)Ga\(_{1-x}\)As [43], and in topological insulators [44, 45]. The constructive interference between time-reversed waves of carriers in a disordered material leads to the weak localization (WL) effect [46]. Applying a magnetic field perpendicular to the plane of motion of carriers inhibits the WL and causes a negative MR at small magnetic fields. A strong SO interaction decreases the probability of carriers backscattering and counteracts weak localization. This effect is called weak anti-localization (WAL) and leads to a positive MR at small fields.

The conduction electrons confined in the vicinity of the polar PAO/STO interface experience a strong electric field perpendicular to the interface as the result of broken inversion symmetry. In the carriers’ rest frame, this electric field appears as an internal magnetic field at the interface plane, perpendicular to their wave vector. As a result, the spins of electrons couple to the internal magnetic field which leads to a large Rashba SO interaction whose magnitude is tunable by the application of an external electric field [23]. We use the same analogy to fit the MR data for our samples. We fit the positive MR data at the \(10^{-5}\) torr sample with the Hikami–Larkin–Nagaoka (HLN) theory [47]. HLN theory considers the effect of SO scattering, random magnetic impurity scattering, magnetic field, and inelastic collisions on the quantum backscattering interference, and is expressed as a change in conductivity:

\[
\Delta \sigma(H) = -\frac{e^2}{\pi \hbar} \left[ \frac{1}{2} \Psi \left( \frac{1}{2} + \frac{H_S}{H} \right) - \ln \left( \frac{H_S}{H} \right) - \Psi \left( \frac{1}{2} + \frac{H_S + H_{SO}}{H} \right) + \ln \left( \frac{H_S + H_{SO}}{H} \right) - \frac{1}{2} \Psi \left( \frac{1}{2} + \frac{H_S + 2H_{SO}}{H} \right) + \frac{1}{2} \ln \left( \frac{H_S + 2H_{SO}}{H} \right) \right] \tag{3}
\]

where \(\sigma\) is the longitudinal conductivity and is calculated from the inversion of the measured resistivity matrix. \(\Delta \sigma(H) = \sigma(H) - \sigma(0)\), \(\Psi(x)\) is the digamma function, \(\frac{e^2}{\pi \hbar}\) is the universal value of conductance and \(H_S = h/4eD\tau_{SO}\). \(D\) and \(\tau_{SO}\) are the diffusion constant and phase coherence time (inelastic scattering time), respectively. \(H_{SO} = h/4eD\tau_{SO}\) is the field at which the SO interaction is no longer effective and the positive WL magnetoresistance becomes the negative WL magnetoresistance. \(\tau_{SO}\) is the SO scattering time. HLN theory was derived for materials in which the spin-splitting energy is proportional to \(k^2\), which is the case in SrTiO\(_3\) [48].
We treated both $H_{SO}$ and $H_\phi$ as fitting parameters and the good quality of the fit in a wide magnetic field range is shown for the $10^{-5}$ torr sample in figure 5. While HLN theory is significant only in the diffusive regime, the fit, however, is shown for a wide magnetic field range. We obtained $H_\phi = 0.33$ T and $H_{SO} = 1.25$ T for perpendicular, and $H_\phi = 0.50$ T and $H_{SO} = 1.25$ T for parallel magnetic field orientations, respectively, for the $10^{-5}$ torr sample.

As the $P_{O_2}$ is increased, the positive part of the MR becomes smaller. The $10^{-4}$ torr sample has positive MR at magnetic fields near $H = 0$. The maximum of its MR happens at 1.5 T. For this sample, we add the WAL positive MR, due to strong SO interaction, to the negative Kondo MR:

$$R(H) = R_0 + R_K \left( \frac{H}{H_1} \right) + c \left[ \psi \left( \frac{1}{2} + \frac{H_\phi}{H} \right) - \ln \left( \frac{H_\phi}{H} \right) \right]$$

where $c$ is a constant and $H_1$ is a magnetic field scale which is related to both $T_K$ and the $g$-factor of the impurity spin. $R_K(H/H_1)$ is the zero temperature Kondo–magnetoresistance (taken at 2 K for our data), which is expressed in [49] as:

$$R_K \left( \frac{H}{H_1} \right) = R_K(H = 0) \cos^2 \left( \frac{\pi}{2} M \left( \frac{H}{H_1} \right) \right).$$

$M(\frac{H}{H_1})$ is the magnetization of a Kondo impurity at zero temperature and is given by:

$$M \left( \frac{H}{H_1} \right) = \left\{ \begin{array}{ll}
\frac{1}{2} \sum_{i=0}^{\infty} (-\frac{1}{2})^i e^{-(i+1)\frac{H}{H_1}} e^{-\frac{1}{2} \Gamma \frac{H}{H_1} (i+1/2)} & H < \sqrt{2} H_1 \\
1 - (\pi)^{-3/2} \frac{\pi}{2} \sin(\pi e^{-1/2}) e^{-1/2} e^{-\frac{1}{2} \Gamma \frac{H}{H_1} (1/2)} & H \geq \sqrt{2} H_1.
\end{array} \right.$$  

This interpretation was previously used by Lee et al \[38\] in electrolyte-gated STO to explain the negative MR regime, and by Das et al \[50\] in $\delta$-doped LaTiO$_3$/SrTiO$_3$ with LaCrO$_3$ to explain the small positive MR which is followed by a transition to a negative MR regime. The result of fitting to equation (4) is shown with the black solid line in figure 4. The WAL effect is meaningful for small fields, $H < H_{SO}$, thus we only fit the data for small field range. The fitting parameters are listed in table 2.

The difference between weak anti-localization in normal and in-plane magnetic fields for thin films contains information about the effective dimensionality of the carrier system. For in-plane fields, if the effective thickness of the carrier system is smaller than the magnetic length (at low fields), then the weak anti-localization magnetoresistance is affected \[51, 52\]. When the field is brought from being perpendicular (figure 4(a)) to being parallel to the interface (figure 4(b)), there is no sign change in the MR values. There is not much change...
Thus, the MR of the 2DEG is smaller than the magnetic length for this interface. For this sample, the 2DEG is thicker than the magnetic length. The positive part of the MR curves for this sample are very similar. Thus, the thickness of the both MR curves for this sample are very similar. Thus, the orientation of the magnetic field: as shown in figure 4(b), the positive part of the MR at low fields becomes smaller for the 10−4 torr sample. We expect that the thickness of the 2DEG is smaller than the magnetic length for this interface. Thus, the MR of the 10−4 torr sample is in accord with the 2D nature of the interface for this material.

4. Conclusion

In summary, we examined the behavior of the conductivity at the interface between PrAlO3 and SrTiO3; we studied the electric and magnetic transport properties of interfaces of PAO/STO grown at oxygen pressures in the P02 range of 6 × 10−5−1 × 10−4 torr. Overall, differences between PrAlO3 and LaAlO3 were subtle, much smaller than the effects of oxygen content changes. These indicate that Pr ions near the interface are predominantly trivalent, with relatively localized magnetism. The most-conducting 2-D-like Pr samples were not as conducting as comparable La samples, indicating either that a steric effect, or a slightly mixed-valent nature of Pr, may be detrimental to conductivity at the interface.

For the 6 × 10−6 torr sample the very small value of R0 at low temperature, large carrier concentration, high mobilities, and very high positive MR values indicate it is like a bulk metal, likely due to extensive oxygen defects and/or interdiffusion.

As the interfaces are grown at higher oxygen pressures, resistivity behavior characteristic of nearly two-dimensional transport occurs; for 10−5 and 10−4 torr, high-temperature metallic behavior with increasing R0 is accompanied by an unjump at low temperature, consistent with Kondo scattering theory. Analysis of the R0 data gives Kondo temperature ~16 K and ~18 K for the 10−5 and 10−4 torr samples, respectively. The MR values for the P02 ≥ 10−5 torr sample was modeled with their positive part due to WAL because of a strong SO interaction and their negative part due to the Kondo effect. The variation of MR values suggests a strong SO interaction for the 10−5 torr sample with HSO = 1.25 T in both field orientations. The positive part of the MR shrinks for the 10−4 torr sample. For this interface the MR is dominated by negative Kondo MR at high fields.

Acknowledgment

The authors acknowledge support from the Robert A Welch Foundation Grant No. F-1191 and from the University of Texas at Austin, College of Natural Sciences Freshman Research Initiative.

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Table 2. Parameters extracted from fitting the MR data in figure 4 to equation (4).

| P02 (torr) | H1(T) | H2(T) | c (Ω) | H1(T) | H2(T) | c (Ω) |
|-----------|-------|-------|-------|-------|-------|-------|
| 10−4      | 11.4  | 0.46  | 800   | 11.6  | 0.52  | 800   |

Figure 5. Dependence of magneto-conductance, expressed in units of e2/πℏ, on magnetic field at 2 K for the 10−5 torr oxygen growth pressure sample for both field orientations. Black lines are fit to equation 3.
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