The emergence of states of matter in low-dimensional systems is one of the most intriguing topics in condensed matter physics. Interfaces between nonmagnetic, insulating oxides are found to give rise to surprising behaviors, such as metallic conductivity, superconductivity, and magnetism.Sensitive, noninvasive local characterization tools are essential for understanding the electronic and magnetic behavior of these systems. Here, the scanning superconducting quantum interference device (SQUID) technique for local magnetic imaging is described and its contribution to the field of oxide interfaces is reviewed.

1. Introduction

Transition metal oxides in the form of a perovskite (ABO₃) are a class of materials that include oxygen and two cations (A and B). Materials of this structure bear potential for future technologies because the spin, charge, orbital, and lattice degrees of freedom in these materials are coupled to each other. Therefore, different perovskites (with different cations) offer a broad range of functional properties, which have been extensively studied in condensed matter physics.

Following the development in deposition techniques of transition metal oxides, it became possible to grow thin films, interfaces, and superlattices with atomic precision. This led to the discovery of low-dimensional states that are not available in bulk oxides, and allowed access to the rich physics of these strongly correlated electron systems.

A prominent example to the emergence of new states at interfaces due to atomic scale changes is the (001) interface between lanthanum aluminate and strontium titanate (LaAlO₃/SrTiO₃, LAO/STO). LAO and STO are both insulating and non-magnetic, but the interface between them exhibits a 2D electron system with high electron mobility, superconductivity, and electric-field-tuned metal–insulator and superconductor–insulator phase transitions. Sample averaging magnetization and magnetoresistance measurements indicate some form of magnetism depending on the preparation conditions, and suggest a tendency toward electronic phase separation.

Other oxide interfaces, like amorphous LAO/STO, γ-Al₂O₃/STO, and LAO/EuTiO₃/STO heterostructures also support interfacial conductivity and superconductivity. However, the origin of these emergent states and their interrelations are not fully understood.

A good way to improve our understanding of the electronic and magnetic behavior in these systems is by using sensitive and local measurement techniques. Mapping the local electronic properties can reveal the nature of the state and explain the electronic behavior of the entire sample or device (the global behavior). The challenge is in utilizing appropriate characterization tools with sufficient sensitivity that do not perturb the measured state.

Various techniques are available for local characterization of surfaces. While local probes like atomic force microscopy, scanning tunneling microscopy, and spectroscopy offer subatomic spatial resolution and extremely high sensitivity to the sample’s topography or to the electronic states at its surface, their effectiveness diminishes when the electronic states are buried under several atomic layers. Therefore, examining electronic states by tracking their magnetic signatures is a useful approach for the case of interfaces.

There are several local magnetic characterization techniques available for studying interfaces between materials, for example magnetic force microscopy (MFM), nitrogen vacancy (NV) center magnetometry, scanning Hall probe microscopy, and scanning superconducting quantum interference device (SQUID) microscopy. The choice of a magnetometer for a specific experiment is influenced by several considerations. First, there is a delicate interplay between the magnetic sensitivity and the spatial resolution: SQUIDs offer excellent magnetic flux sensitivity often with limited spatial resolution, while MFM offers a spatial resolution of 10 nm, and NV centers can reach 1 nm spatial resolution, together with excellent magnetic field sensitivity. Other considerations involve, for example, accessible temperatures: MFMs and NV centers operate at temperatures ranging from milikelvins to 300 K while SQUIDs function well at milikelvins, but cannot operate at room temperature. Finally, one should also consider the invasiveness of each technique. For example, MFMs apply large magnetic forces on the sample, which are necessary for the imaging. MFM were previously used to manipulate magnetic objects such as vortices. SQUIDs and NV centers on the other hand apply a negligible magnetic field to the sample. This may be important when looking for small traces of magnetism, which might be perturbed by the sensor.

Here, we focus on the information that was gained by utilizing scanning SQUID technique to investigate several oxide interfaces. Scanning SQUID can simultaneously map electronic and magnetic properties. We show that exciting physics can be revealed by scanning SQUID tracking of the local variation of electronic properties.

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2. Scanning SQUID for Noninvasive Mapping of Local Magnetic Flux

SQUID is a highly sensitive magnetometer based on a superconducting loop interrupted by two Josephson junctions. The SQUID measures small changes to the magnetic field that threads the superconducting loop (magnetic flux) by utilizing the sinusoidal flux–voltage relation caused by the two junctions. SQUIDs can resolve extremely small changes to the magnetic flux, smaller than 1 \( \mu \Phi_0 \) \((\Phi_0 = h/2e = 2.07 \times 10^{-15} \text{ Wb})\), the magnetic flux quantum.

SQUIDs can be fabricated in a variety of sizes, ranging from centimeters to 40 nm. For scanning applications, where both high spatial resolution and magnetic field sensitivity are desired, there is a delicate tradeoff between the two. For a given flux sensitivity of 1 \( \mu \Phi_0 \), the magnetic field sensitivity depends on the size of the loop. For example, a loop with 1 \( \mu \)m radius would yield a magnetic field resolution of \( \approx 1 \text{ nT} \), while a radius of 50 nm yields resolution of \( \approx 1.5 \mu \text{T} \).

SQUIDs have been used as sensitive magnetometers for decades, and in the 1980s, as lithography capabilities developed, the planar SQUID with an integrated pickup loop was developed and since then continued to evolve (Figure 1a). In order to achieve local sensitive measurements with a SQUID, the sensitive area (the closed loop that captures the field lines) needs to be as small as possible and be brought close to the surface of interest. For best sensitivity the proximity to the sample needs to be similar to the diameter of the loop. In addition, tuning the physical properties of the SQUID—inductance, capacitance, resistance and the geometry of the Josephson junction—improves its noise characteristics.

There are two ways to achieve high spatial resolution for SQUID sensors. One approach is to create small SQUID devices and use them as sensors. Scanning SQUIDs were successfully fabricated on a quartz tip by Finkler et al. by evaporating the superconductor from the sides of the tip, creating a small loop at the apex (Figure 1b). The loop is thicker at the leads, and this way the loop has two constriction Josephson junctions. In this geometry, the SQUID-on-tip can be brought close to the sample surface and reach spatial resolution of few tens of nanometers. In addition, the small area of the SQUID and leads makes it possible to apply magnetic fields up to a Tesla during the measurements. The critical current is very sensitive to temperature, and this allowed the development of a unique thermal dissipation imaging with sensitivity of 1 \( \mu \text{K} \).

The second approach is to keep the SQUID large and fabricate a small sensing loop that extends far away from the SQUID itself. This is a rather large SQUID circuit, about a millimeter, with a small pickup loop that is separate from the core area of the SQUID that includes the junctions, junction shunts, and modulation coils (see Figure 1a). The size of the pickup loop ranges from few hundred nanometers to few micrometers. The SQUID is prepared by a multilayer process, with tunnel junctions and the ability to control and optimize SQUID properties, implement a good shielding scheme, and add on chip electronics. These SQUIDs can be made in a gradiometer configuration to eliminate background fields, and

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Figure 1. Scanning SQUID imaging. a) Planar SQUIDs. (Left) Optical picture of a micro-SQUID, zoomed in to show the pickup loop and field coil. (Middle) Depth resolved image of a 600 nm pickup loop and field coil, and (right) schematic sketch of the SQUID’s circuit. (Middle) Adapted with permission. Copyright 2008, American Institute of Physics. (Left) Adapted with permission. Copyright 2008, American Institute of Physics. b) SQUID-on-tip. (Left) A sketch of the tip. The dark gray area is superconducting. (Right) Scanning electron microscopy image of the edge of the tip. (Left) Adapted with permission. Copyright 2013, Springer Nature. c) SQUIDs can simultaneously map different electronic properties. Both SQUID-on-tip and planar SQUIDs map the ferromagnetic landscape and current flow in different materials. Planar SQUIDs use an integrated local field source to map the local susceptibility, and the SQUID-on-tip can be used as a temperature sensor, mapping thermal dissipation.
includes an on-chip modulation coil as a feedback circuit to maximize sensitivity by operating the SQUID in a flux locked loop. In addition, the multilayer approach allows to add the capability to image local susceptibility, simultaneously with the magnetism, by including a local field source.\textsuperscript{[37]}

The physical properties that can be imaged using SQUIDs are illustrated in Figure 1c. The scanning SQUID can: (i) map the landscape of static magnetic flux, (ii) detect the magnetic response to current flow by sensitive noninvasive mapping of the magnetic field generated by the current flow, (iii) image local susceptibility and this way track the paramagnetic response to localized spins, (iv) map very small traces of superconductivity using the local diamagnetic response, even before the superconductivity percolates all the way to the lead at the other side of the sample, and (v) map tiny quantities of thermal dissipation. The properties can be measured simultaneously, which enables studies on correlations between the different physical properties.

For (i)–(iv) the SQUID measures the flux that threads the pickup loop. Since the magnetic field lines spread out quickly when moving away from the source, the distance between the sensor and the sample is an important factor in determining the spatial resolution of the sensor. For optimal measurement conditions, the sensor–sample distance, the size of the loop, and the size of the feature should be similar. The measured flux can then be converted to magnetic field units, by deconvoluting the flux with the point spread function (PSF) of the sensor. The deconvolution also requires knowledge of the sensor–sample distance. The details of the PSF, which may be complicated for a real measurement,\textsuperscript{[47]} can be independently measured, while the sensor–sample distance is not always accurately known. In the following sections most of the SQUID data are presented in units of flux, and we estimate the magnetic field signal by dividing the flux data by the sensitive area of the SQUID.

3. Local View of Emergent Electronic Behaviors at the Interface

Local view of the magnetism at the LAO/STO interface was first achieved by Bert et al.\textsuperscript{[48]} The magnetism appears in the form of isolated ferromagnetic patches, inhomogeneously distributed throughout the sample. To track the origin of the magnetism, Kalisky et al.\textsuperscript{[49]} examined the ferromagnetism as a function of the LAO layer thickness. Surprisingly, they observed ferromagnetic patches only in samples with LAO thickness exceeding 3 uc (Figure 2a), close to the critical thickness for conduction at the interface, supporting an electronic reconstruction scenario for the ferromagnetic phase.

While a critical thickness is required for both the conduction and the magnetism to appear, magnetism was observed also in SrO terminated LAO/STO,\textsuperscript{[46]} which does not display interfacial conductivity.\textsuperscript{[6]} This shows that conduction is not necessary for the ferromagnetic state to exist. Another evidence that separates the observed magnetism from the conductivity comes from gating experiments in TiO\textsubscript{2} terminated LAO/STO where unlike the conductivity, the magnetism was insensitive to the electric field.\textsuperscript{[50]} Another difference is that while the conductivity reliably appears in all samples above the critical LAO thickness, some samples (grown in identical conditions) show no trace of magnetism.\textsuperscript{[49,51]} These results suggest that the electron population that participate in the ferromagnetic order is different than the one that participates in conduction.

The ferromagnetic patches were further studied applying local stress. Kalisky et al.\textsuperscript{[52]} found that local stress applied to the patches by the tip of the sensor may cause variations to the dipole moments and their orientation. A possible scenario is that the patches are comprised of smaller, resolution-limited ferromagnetic domains that are reoriented by stress.

By cooling the samples below $\approx$300 mK, LAO/STO becomes a superconductor. Bert et al.\textsuperscript{[48]} locally studied the superconducting phase at the LAO/STO interface at 30 mK. Utilizing the on-chip field coil they mapped the local diamagnetic response from the superconductor and found a highly spatially inhomogeneous response (Figure 2b). Over length scales of few tens of micrometers the signal varied by 84%, compared to 12% variations observed in the superconducting $\delta$-doped STO. Above the superconducting transition, the samples were found to host homogeneous paramagnetism.

The ability to simultaneously map both susceptibility and magnetometry made it possible to study the interplay between the two seemingly opposing phenomena of superconductivity and ferromagnetism. Bert et al. found that ferromagnetic patches coexist with the superconducting phase, but without correlation between their spatial distributions, supporting the scenario of phase separation.\textsuperscript{[48]}

Simultaneous measurements of different electronic phases help us relate to the existing theoretical predictions, since we can estimate the number of electrons that participate in each electronic phase. For example, the coexistence of the different mobile and localized electronic phases could resolve the problem of the missing electrons in the polar catastrophe scenario.\textsuperscript{[53]} Transport experiments found carrier densities\textsuperscript{[8,9,12]} that are one order of magnitude smaller than the density predicted by the theory.\textsuperscript{[53]} Extracting the number of electrons that participate in each phase suggested that the missing electrons are localized, and therefore do not participate in the conductivity.\textsuperscript{[48]}

The superfluid density in LAO/STO can be tuned by an electric field applied between a back gate and the conducting interface.\textsuperscript{[8]} Scanning SQUID measurements as a function of the gate voltage followed the changes in the magnitude and the spatial distribution of the diamagnetic response (Figure 2c).\textsuperscript{[50]} The local diamagnetic signal reflects the Pearl length,\textsuperscript{[54]} the penetration depth of this quasi-2D superconductor, and its temperature dependence yields information about the order parameter. The temperature evolution of the local diamagnetic response, measured at different gate voltages, collapsed to a single curve. This collapse indicates that throughout the superconducting dome, with peak at 240 mK, the superconductivity can be described by the Bardeen–Cooper–Shrieffer theory.\textsuperscript{[50]} The conductivity, superconductivity, and magnetism described above were all measured in LAO/STO. Another interface whose magnetic behavior has been investigated with the SQUID microscope is between LaMnO\textsubscript{3} (LMO) and STO. In bulk, underdoped LMO is an insulating antiferromagnet, but at high doping or at high oxygen content, bulk LMO is
Figure 2. Scanning SQUID studies of emergent magnetism at oxide interfaces. a–d) LAO/STO. a) Maps of the magnetic landscape of samples with varying LAO thickness, revealing that ferromagnetic patches appear only above a critical thickness. Flux of 30 m\(\Phi_0\) is equivalent to \(\approx 30 \mu T\) for the sensors used in this study. Inset: The measurement scheme, showing the SQUID’s pickup loop near an inhomogeneous magnetic landscape. Adapted with permission.[49] Copyright 2012, Springer Nature. b) Magnetic susceptibility map taken at 40 mK revealing spatial inhomogeneities in the superconducting landscape (blue indicates stronger superconductivity). Flux is normalized by the current in the field coil. 0.5 \(\Phi_0 \ A^{-1}\) convert into \(\approx 0.5 \mu T \ A^{-1}\). Inset: The measurement scheme. The larger loop applies a local magnetic field and the smaller pickup loop records the sample’s response. Adapted with permission.[48] Copyright 2011, Springer Nature. c) Magnetic susceptibility maps taken at different back gate voltages, from the insulating phase (left; \(V_{BG} = -70\) V) through the superconducting phase. Inset: The measurement scheme. Adapted with permission.[50] Copyright 2012, American Physical Society. d) Top: The magnetic field from an individual ferromagnetic patch after repeated scans in contact. The dipole moment changes its orientation between the scans. Bottom: A sketch showing the silicon chip (blue) pressing the sample, and the SQUID’s pickup loop (red) detecting the magnetic response. Adapted with permission.[52] Copyright 2012, American Chemical Society. e–g) LMO/STO. e) Maps of the magnetic landscape of samples with varying LMO thickness, revealing that a magnetic state only appears above a critical thickness. Colorbar spans linearly: in the two left panels \([A,B] = [-2,2] \mu T\), in the two right panels \([A,B] = [-20,20] \mu T\). Adapted with permission.[57] Copyright 2015, The American Association for the Advancement of Science. f) Images of the magnetic landscape in the zero field cooled (ZFC) state (left), and with applied in-plane magnetic field of +250 mT (middle) and -250 mT (right). In the latter two the polarity is switched, demonstrating domain flipping. Colorbar spans linearly: In the leftmost panel \([A,B] = [-1.5,1.5] \mu T\), in the two right panels \([A,B] = [-1.8,1.8] \mu T\). Adapted with permission.[58] Copyright 2016, Springer Nature. g) An illustration of the microscopic magnetic state, consisting of superparamagnetic (SPM) LMO islands embedded in an antiferromagnetic (AFM) matrix. Adapted with permission.[58] Copyright 2016, Springer Nature.
ferromagnetic and metallic.\cite{55} In thin films, stoichiometric LMO is ferromagnetic and insulating.\cite{56} Since LMO, like LAO, is polar, electronic reconstruction is predicted in LMO/STO after a critical thickness, similar to the LAO/STO interface.

Scanning SQUID measurements by Wang et al. revealed a thickness-dependent magnetism in LMO/STO.\cite{57} They observed a sharp ferromagnetic transition above a critical thickness of 6 uc of LMO (Figure 2e), with highly inhomogeneous magnetic landscape. Below the critical thickness no magnetic signal was observed, suggesting that the LMO is antiferromagnetic. Another confirmation to the orbital reconstruction mechanism was that nonpolar CaMnO3 grown on δ-doped-STO did not show a similar behavior.\cite{57}

Anahory et al. used scanning SQUID-on-tip to study magnetism in LMO/STO.\cite{58} They observed a magnetic phase above the same critical thickness and identified the magnetism as a superparamagnetic phase. Mapping the magnetic landscape in the presence of an external in-plane magnetic field, Anahory et al.\cite{58} showed that magnetic islands reverse their magnetization (Figure 2f). This is different from the behavior of a ferromagnetic sample, where applying external field would enlarge domains oriented with the field and shrink domains oriented against it. This indicated that the magnetic phase is superparamagnetic, in which small ferromagnetic islands are embedded in a large antiferromagnetic matrix (Figure 2g).

Another demonstration of the usefulness of scanning SQUID in mapping magnetism at oxide interfaces are measurements of SrRuO3/STO. SrRuO3 is highly conductive and ferromagnetic.\cite{59,60} However, thin films of SrRuO3 below three unit cells thick, were shown to be insulating,\cite{61,62} and some theories predict an antiferromagnetic order.\cite{63} When a single unit cell of SrRuO3 is grown on STO, it is predicted to host a conductive, spin polarized phase.\cite{64,65} This phase is only predicted below 105 K, when STO undergoes a structural phase transition. To test this hypothesis, Hughes et al.\cite{66} fabricated high quality (SrRuO3)1-(SrTiO3)δ superlattices that exhibit a metallic behavior. Scanning SQUID imaging of the magnetic landscape of the samples revealed a complex magnetic domain configuration, proving the existence of a ferromagnetic phase as well.

4. Correlations between Lattice Structure and Electronic Properties

Local mapping by scanning SQUID gained valuable information about electronic states in oxide interfaces. Electrons in 2D interfaces are strongly influenced by both ordered and disordered constraints that may originate in the structure of the substrate or local disorder. Mapping the spatial distribution of electronic properties can teach about the way electrons interact with local physical conditions, which can modulate due to the substrates structure, disorder, phase transition of a parent compound, local strain, or even other electron populations.

In 2013, Kalisky et al.\cite{67} imaged the normal current flow at the LAO/STO interface. Strikingly, they found that the current flow is modulated over stripes, directed along the STO's crystallographic directions (Figure 3a). The modulation changed upon thermal cycles above 105 K, the cubic-to-tetragonal structural transition of STO, suggesting that the modulations in the current flow are over the domain structure. In the superconducting phase, the diamagnetic response, reflecting the superfluid density, was modulated over the same structure.\cite{67}

This work led to further studies, due to the multiple questions it raised regarding the origin of the modulations, which were not expected in the quasi-2DEG. One question was whether this stripy inhomogeneity could affect transport measurements of an entire sample or a device. The possibility of correlations between the crystal structure and other electronic properties of the LAO/STO interface, as well as of other STO-based systems, is also intriguing questions.

The domain structure in the tetragonal phase of STO is well known,\cite{68,69} and since a new configuration of domains appears upon thermal cycles around the transition temperature, global properties can change between cooldowns. The relation between global measurements and SQUID images was investigated by Frenkel et al.\cite{70} by mapping the spatial distribution of the current flow with the SQUID, while simultaneously measuring the global transport values. For devices that are few hundred micrometers, which is smaller than the typical domain wall length, the domain wall configuration caused dramatic changes to the resistivity. The anisotropy of the resistivity measured by transport in Van der Pauw devices was large, and the local view identified these as cases where the domains were all aligned in the same direction. By combining a scanning stress microscopy with scanning SQUID, Frenkel et al. recently found that the origin for the effect STO domains has on conduction at the interface is that STO domain walls are polar (Figure 3b,c).\cite{71}

Investigating the superconducting phase in δ-doped STO, Noad et al.\cite{72} found spatial variations of the superconducting critical temperature. By measuring local diamagnetic response as a function of temperature, they found areas with increased critical temperature, up to 10% higher than the temperature where the entire sample was superconducting. While the changes appear along stripes in the crystallographic directions of the substrate, their width varies and can be tens of micrometers wide, too wide to be attributed to domain walls (Figure 3d), leading to the conclusion that the variations of the critical temperature are related to differences between properties of adjacent domains. The dielectric constant of STO may change by over a factor of two between different domains\cite{73} and may be the origin of the critical temperature variations between them.

An interesting question is whether STO domain walls can affect other systems grown on STO, where the superconductivity is not interfacial. Wissberg and Kalisky\cite{74} observed similar modulations in other superconducting systems (polycrystalline Nb, NbN, and epitaxial YBa2Cu3O7−δ grown on STO. Scanning SQUID maps of the local diamagnetic response found weak, large scale, stripy modulation along the STO crystallographic directions in all films (Figure 3e). The signal variations over the stripes strongly increased when approaching the critical temperature. Above the critical temperature, in the normal state, SQUID maps of the normal current flow showed weak modulations along the same stripes.\cite{74} These features were observed in superconducting layers with thicknesses up to 100 nm. These results show that the effects of the STO substrate are more general, extending to systems where conductivity is not confined to a thin nanometric layer at the interface.
Another property that may be modified by the structural domains of STO is magnetism. Rosenberg et al. studied the magnetic landscape of epitaxial ferromagnetic EuS thin films grown on STO. Upon cooling the sample below the Curie temperature in the presence of an in-plane field, the authors observed modulations of the magnetic field over stripes along the STO’s crystallographic directions (Figure 3f). Thermal cycles above 105 K confirmed that the stripes change their...
configuration. The authors suggest local strain variations due to the tetragonal domain walls as the cause for the modulation, but further studies are required to confirm this explanation.

5. Conclusions and Future Directions

We described the scanning SQUID technique, and the physical properties that it can map. The unique view provided by the SQUID allows sensitive and noninvasive investigation of electronic properties via tracking small magnetic fields. We described recent scanning SQUID studies of oxide interfaces. The amount of valuable information that was gained in the past 6 years demonstrates the power of this technique.

We showed the variety of electronic degrees of freedom, which coexist or spatially modulate at oxide interfaces. This richness raises fundamental questions about the nature of the 2D system at the interface, and opens doors to technological applications.

One open question is the nature of the magnetism observed in LAO/STO. Many theoretical models were offered to explain the elusive magnetism at the interface, but additional, careful local characterization of the magnetic landscape at the interface is required to reach a conclusion.

Another question is related to the conduction over STO domain walls, which seems to be different than the flow in the bulk. Could the domain walls host 1D channels for the current? If so, what about the superconductivity at the domain walls? These questions can be addressed by combining local imaging techniques with transport measurements.

On the technological side, the presence of polar domain walls, whose configuration can be controlled by either thermal cycles or electric fields, suggests using the domain walls as local gates in an oxide-based circuit. For example, a domain wall that interrupts a superconducting LAO/STO ring could serve as a Josephson junction, thus creating an SQUID. An LAO/STO-based SQUID was successfully achieved by fabricated local gates. Another possible future application is changing the configuration of the domain walls (i.e., by applying a back gate voltage), in order to tune the properties of a circuit. For the development of such dynamic electronics, the control over domain walls configuration should be improved, and local tools must be involved in the development and characterization.

We expect that SQUIDs will continue to serve as one of the prominent research tools for the study of oxide interfaces, and continue to supply the oxides community with exciting observations that will contribute to the development of the field.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

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