	Experiment title:	Experiment number:
ESRF	Study of structural modification at the field effect induced metal-insulating transition of the $SrTiO_3$//$LaAlO_3$ interface by Grazing incidence X-ray Diffraction	SI1853

Beamline:	Date of experiment:	Date of report:
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Shifts:	**Local contact(s)**:	*Received at ESRF:*
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Report:

Interfaces between perovskite oxides may exhibit unexpected physical properties that depend drastically on the mutual arrangement of each single atomic plane. Outstanding examples come from the combination of band and/or Mott insulators, in particular in (001) $LaTiO_3/SrTiO_3$ multilayers [1] and $LaAlO_3/SrTiO_3$ heterostructures (LAO/STO) [2]. It has been proposed that interface phenomena dominate the characteristics of these systems :a case study is the realization of a **2D conducting electron gas** at the TiO_2/ LaO ntype interface (IF). However, the issue is a subject of an animated debate, and especially in the case of the LAO/STO IF, no consensus nor a clear microscopic picture for the electronic state has been reached. The original mechanism proposed to explain the phenomena is related to the "polar catastrophe" idea. The LAO/ STO system is composed by alternating charged atomic planes, LaO^+ and AlO_2^- layers, on top of a neutrally charged TiO_2 surface terminating the STO single crystal. In this scenario, above the critical thickness of four LAO unit cells, the electrostatic energy accumulated is released by transferring half electron per unit cell at the interface. These surface carriers would fill the originally empty Ti 3d band and realize an electron gas confined just at the interface [2]. The electron reconstruction could eventually be accompanied by a structural reconstruction.

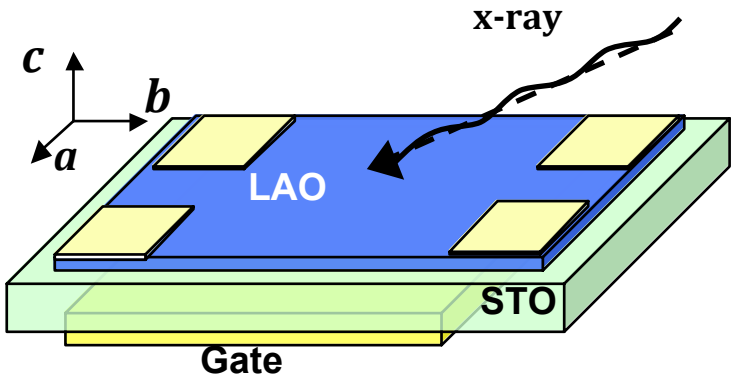

Fig. 1: Sketch of the experimental set-up used during the experiment. The four contacts connected to the ground while the gate will be connected to a 6517A electrometer/voltage source.

The transition from an insulating to a metallic interface can be attained also by using the electric field effect [3, 4]. In particular an insulating to metal transition is achieved on samples composed of 3 unit cell of $LaAlO_3$, which have the critical thickness required to observe the electron gas.

In the SI1853 experiment we have attempted to perform a crystallographic refinement of the interface structure of the LAO/STO system, across the insulating to metal transition induced by the electric field effect. Grazing Incidence X-ray diffraction is a quantitative technique able to give precise information about the atomic displacements and cation substitution at the surface/interface of single crystals.

High quality LAO/STO heterostructures have been realized at the University of Augsburg and University of Geneva by Pulsed Laser Deposition and *in-situ* monitoring by reflective high-energy electron diffraction will be used to achieve an atomic layer control of the growth [3, 4]. The samples have been fully characterized by AFM, X-ray and transport measurements using laboratory equipments.

In order to study samples with controlled transport properties, eventually modified by the electric field, we have modified the ID03 sample holder in order to allow direct four probe measurements of the sample resistance and to allow the application of an additional gate voltage on the back of the STO single crystal on which the LAO films are deposited, which served also as a gate dielectric because of its high dielectric constant [3, 4]. We have tested the new sample holder mounted on the UHV end-station for GXID measurements of ID03, by measuring *in-situ* the resistance of 3uc and 4uc LAO/STO samples, which are respectively insulating and conducting without the application of an electric field [Fig.1]. At room temperature, and without the beam on the sample, the expected modulation of the conductivity of the 4uc LAO/STO sample was observed, while it was not possible to obtain an insulating to metal transition in the 3uc sample. Then we performed a complete structural refinement of conducting 4uc sample using GXID at room temperature. In Fig. 2 an example of CTR's measured on this sample, together with a fit using a model for the LAO/STO structure is presented.

We tried to obtain a similar structural refinement at low temperatures with and without the electric field applied on the 3uc sample. By lowering the temperature, we were able to measure its resistivity with the beam on or off. It was clear then that the high energy x-rays used (17 keV) modify the conductivity of the interface by creating photo-generated carriers even in insulating 3uc samples. This result made impossible a a complete discernment of the electric field effect on the conductivity of the sample at low temperatures, since it was largely dominated by the photo-generated carriers. Even by closing the beam, the photo-generated conductivity lived for long time (hours).

The experiment has been completed by performing a complete structural refinement of an insulating 2uc LAO/STO sample at room temperature.

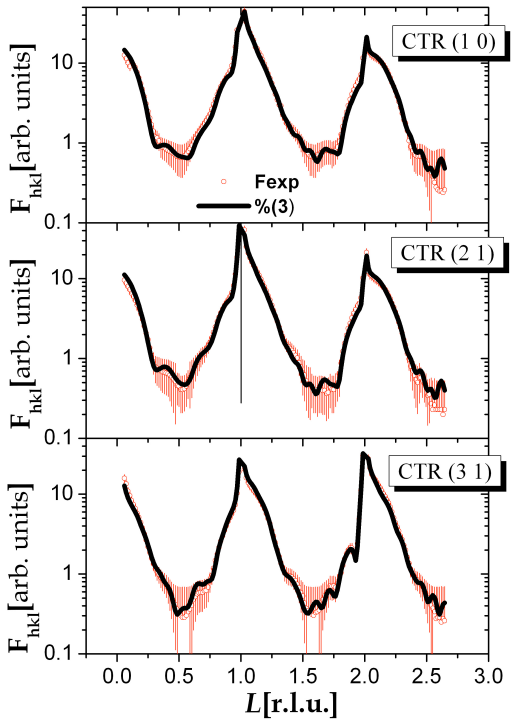

Fig.2: Some CTR'S of a 4uc LAO/STO sample. The black-line is a fit of the data using a 1x1 model.

The analysis of the experimental data, which is in progress, indicate that the electronic reconstruction of the LAO/STO interface is accompanied by a structural and chemical reconstruction of the interface.

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[2] A. Ohtomo and H. Y. Hwang, A high-mobility electron gas at the $LaAlO_3/SrTiO_3$ heterointerface. *Nature* **427**, 423 (2004).
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[4] A. D. Caviglia, S. Gariglio, N. Reyren, D. Jaccard, T. Schneider, M. Gabay, S. Thiel, G. Hammerl, J. Mannhart, J.-M. Triscone, Electric field control of the $LaAlO_3/SrTiO_3$ interface ground state. *Nature* **456**, 624 (2008).