STO/LAO sample Preparation for X-Ray standing Wave Measurements

/ CHRISTOPH SCHLUETER (1,*), JÖRG ZEGENHAGEN (1)

(1) ESRF European Synchrotron Radiation Facility, 6 rue Jules Horowitz. BP 220, 38043 Grenoble (France)

INTRODUCTION.

The formation of a quasi-2dimensional electron gas at interface of $SrTiO_3$ (STO) and $LaAlO_3$ (LAO) attracted considerable attention in the recent years. (Ohtomo, 2004) Although it is generally agreed that the phenomenon is induced by subtle structural changes, the mechanisms are still hotly discussed. So far several explanations for this phenomenon have been proposed, in particular doping with electrons (Pentcheva et al., 2006; Thiel et al., 2006), oxygen vacancies (Kalabukhov et al., 2007), interdiffusion (Nakagawa et al., 2006 ; Willmott et al., 2007), and lattice distortions.

A thorough X-ray standing wave (XSW) and X-Ray Photoemission (XPS) analysis may help to elucidate details of the electronic and atomic structure at the interface. Samples of high crystallographic quality are needed for these experiments. The progress in sample preparation and characterization will be presented.

SAMPLE PREPERATION.

Samples were grown on (001) surfaces of edge oriented $SrTiO_3$ single crystal substrates. To obtain TiO_2 terminated surfaces the substrates were ultrasonically soaked in deionized water for 10 min and subsequently etched in a commercially available buffered HF solution (Merck) for ~30s. Soaking in water leads to a hydroxylation of the SrO sites and thereby facilitates the etching process. The surfaces recrystallize during a final 1h annealing step at 950°C in 1bar molecular oxygen flow. (Koster et al., 1998) Substrates were first cleaned with ethanol and then acetone in an ultrasonic bath for several minutes before introduction into the UHV chamber.

The thin films were deposited in a UHV compatible pulsed laser deposition (PLD) chamber using a (Lambda Physics) Compex 205 UV laser (λ=248nm). Substrates were annealed at deposition temperature and exposed to the oxygen atmosphere for several minutes prior to the deposition. Temperatures of 750 - 820°C and an oxygen atmosphere of 5×10^{-5}mbar were chosen in accordance with the literature. One Laser pulse was found to be equivalent to ~0.7Å LAO by calibration with reflectivity measurements. The target substrate distance was set to 5cm. Some films were subjected to a tempering step at deposition temperature for up to 15 min. After deposition the films were cooled in the deposition atmosphere to room temperature.

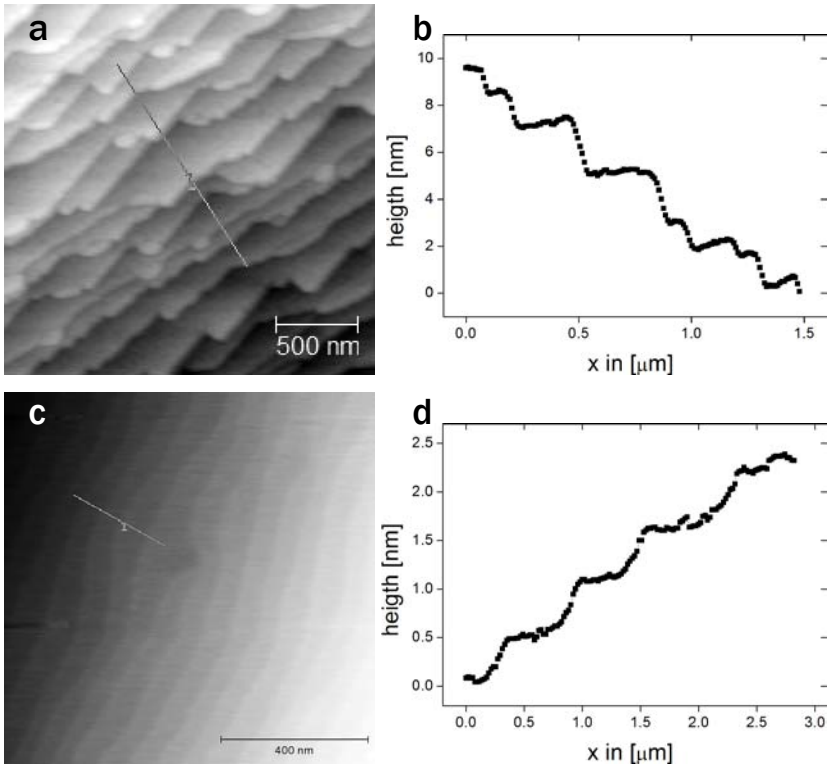

fig 1. AFM images and profiles of ~8uc thick LAO films deposited at 820°C. The cubic axes are rotated by 45°to the scanning direction. (a) tempered for 10min, cooling time 2h with corresponding profile (b) showing several nm steps. (c) 1h cooling time and no tempering. The line profile (d) reveals ~0.4nm unit cell steps.

RESULTS.

The effect of cooling time and a tempering step at deposition temperature were studied systematically by Surface-XRD, reflectivity and AFM experiments.

Reciprocal space mapping showed the formation of powder rings for samples that were subjected to fast cooling (<1h).

palabras clave: LAO, STO, Heterostructura, PLD, Perovskita.

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* corresponding author: schluete@esrf.fr

Samples cooled to room temperature directly exhibit unit cell size steps (lattice constant LAO a=3.9Å) under the AFM. Consequently the terrace width is reduced to a few tens of nm. The edges show no crystallographic orientation.

Samples held at elevated temperatures for longer periods (tempering and/or slow cooling) exhibit several hundred nm wide terraces and step heights of up to 3nm in the AFM (Fig 1 a, b). Many step edges form 90° angles. The scan direction was rotated by 45° to the crystallographic axes. Therefore it can be seen that most edges follow the cubic [001] and [010] directions.

Reflectivity was measured for the same sample (Fig 2). The experimental data was fitted with a simple one layer model having a sharp STO/LAO interface (roughness 1.25Å) and a slightly rougher surface (roughness 3.5Å).

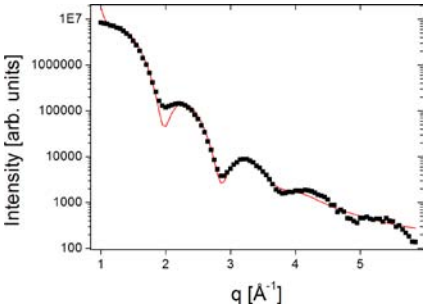

fig 2. *A representative reflectivity scan of the ~8uc thick LAO film.*

Diffraction experiments confirm the good crystallinity and epitaxy of the overlayer.

CONCLUSIONS.

The results presented show that well ordered epitaxial films with sharp interfaces can be grown by the described procedure.

For sample held at elevated temperatures for longer times (tempering and/or slow cooling rate) steps were found to bunch. This is indicative on a significant surface diffusivity at these temperatures. It could not be distinguished whether the bunching is driven by the attempt to minimize the number of energetically unfavorable kink sites or to compensate for strain induced by the lattice mismatch.

Faster cooling times (1-1.5h) resulted in smooth surfaces with unit cell steps. For even higher cooling rates powder rings indicate that parts of the film are randomly oriented.

Investigations of the electronic properties of the films are in progress.

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