

## Transport in $\text{LaTiO}_3 / \text{SrTiO}_3$ nanodot arrays

### $\text{LaTiO}_3 / \text{SrTiO}_3$ ナノドットアレー構造の輸送特性

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#### Introduction

It is well known that charge redistribution occurs at interfaces in semiconductor heterostructures. In oxide heterostructures, the charge transferred across an interface can be large enough to induce metallic conductivity in otherwise insulating materials, raising the possibility of constructing two-dimensional electron gas (2DEG) systems in strongly-correlated materials. The depth of charge spread at the interface is an important factor that determines the dimensionality of the conducting layer. However, it is difficult to measure directly the charge transfer length, partly because it is necessary to measure local carrier densities of about  $10^{17} \text{ cm}^{-3}$ . So far, direct measurement of electron accumulation profiles has been done by electron energy loss spectroscopy (EELS) for  $\text{LaTiO}_3 / \text{SrTiO}_3$  (LTO/STO) interfaces and the accumulation layer thickness is known to be a few nanometers.[1]

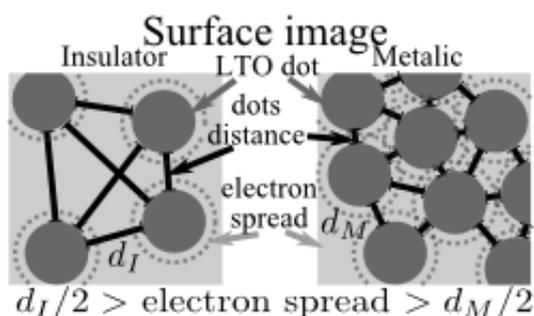


Fig. 1 Dot array becomes metallic when the average dot distance is smaller than the charge transfer length. The purpose of this work is to measure the lateral spread of charge close to the edge of epitaxial nanostructures.

#### Experiment

The purpose of the experiments was to determine the critical coverage, nanodot size, and average dot distance for various nanodot arrays, and to compare the structural parameters with the transport data. Nanodot array samples were grown in pairs, with one array grown on a non-doped  $0.2^\circ$  miscut STO(001) substrate for transport measurements and another equivalent sample grown on a  $0.2^\circ$  miscut 0.05 wt% Nb-doped STO(001) substrate for scanning tunneling microscopy (STM) and atomic force microscopy (AFM) measurements. The samples used for transport measurements were also capped with a STO film to reduce the influence of surface depletion on the transport data. Both types of substrates were wet etched and annealed at  $1000^\circ\text{C}$  and  $10^{-5}$  Torr for 10 minutes before deposition, giving regular step-and-terrace surfaces with an average terrace width of about 150 nm. All films were grown by pulsed laser deposition (PLD). The LTO nanodots were grown at pressures below  $10^{-4}$  Torr to avoid the formation of the  $\text{La}_2\text{Ti}_2\text{O}_7$  phase. [2] The deposition rate was measured by a stylus profilometer for thick films and by monitoring in-situ reflection high-energy electron diffraction (RHEED) specular intensity oscillations during deposition. The growth rate could be adjusted by changing the pulse energy of the ablation laser. The nanodot sizes were controlled by systematically varying the coverage of the LTO

layer and the nanodot growth temperature. Since the surface migration rate of adatoms is a strong function of temperature, it is easy to adjust the average dot size on the 1 to 100 nm scale. Samples used for transport studies were also post-annealed in air at 400°C for 6 hours to eliminate oxygen vacancies. The STM measurements were done with a tungsten tip in vacuum at a base pressure of  $5 \times 10^{-9}$  Torr.

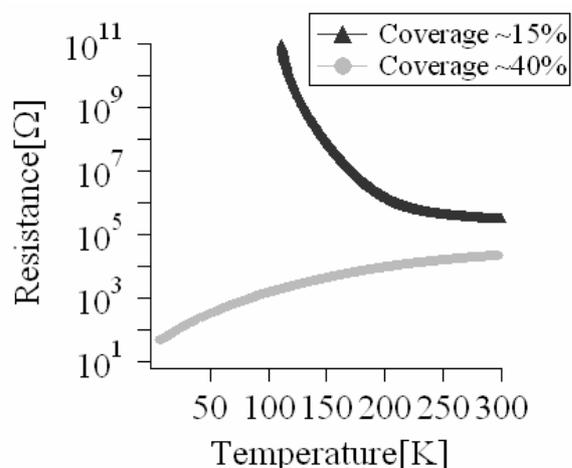


Fig3. Resistance of nanodot arrays grown at 800°C with coverages of ~15% and ~40%.

## Result and Discussion

### Dot distance estimation

The LTO nanodot size depends on the growth temperature. Due to the limited spatial resolution of AFM, the dot diameters could only be reliably analyzed for samples grown at 800°C or 900°C. At 900°C and higher, step-flow growth started to occur due to the large diffusion length of surface atoms. This resulted in the growth of nanowires along the surface step edges in addition to the dots. A growth temperature of 700 to 800°C was thus found to be optimal, as it results in dots that can be easily analyzed by AFM or STM, but nanowire formation does not occur.

The resistance measurement results for two dot arrays grown at 800°C are shown in Fig. 3. It is clear that at low dot coverage, the sample is

insulating. Although the STO substrate becomes metallic underneath the LTO dots, a conduction path does not appear at this coverage, as the average distance between the nanodots is larger than the charge transfer length. Metallic behavior is seen in a sample with 40% coverage. In this case, the LTO dots are still separated structurally, but the dot array shows metallic conductivity. This shows that the electronic size of the nanodots is larger than the physical size. STM images of nanodot arrays grown at the same temperature and with the same coverages are shown in Fig. 4.

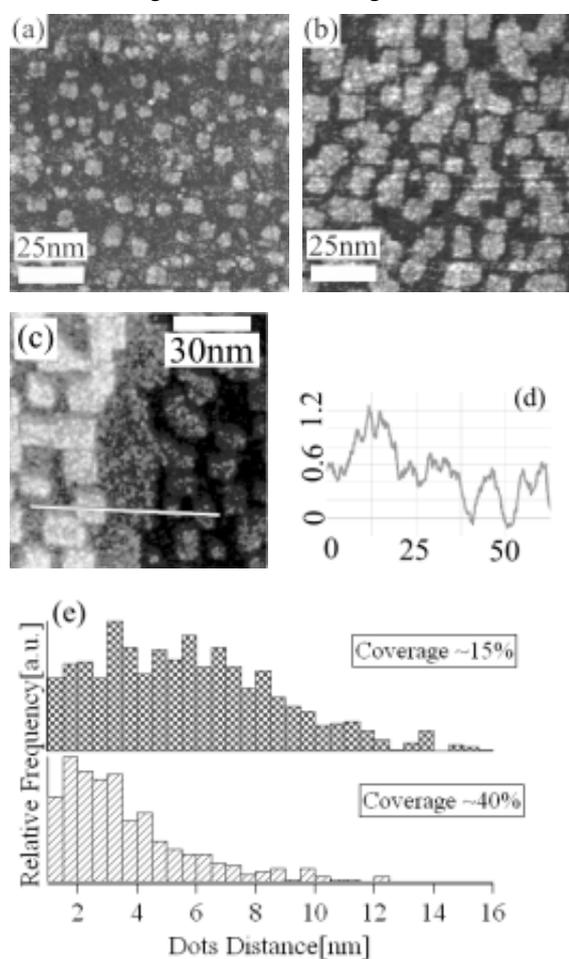


Fig4. STM image of nanodot arrays grown at 800°C with coverages of (a) ~15% and (b) ~40%. Dot image around step edge (c) and its height profile (d). Dot distance histogram (e)

The dot coverages estimated from Fig. 4 are (a) 21% and (b) 45%. The difference between the RHEED and STM coverage estimations can be

caused by the inaccuracy of the RHEED oscillation measurement. Figs.4 (c) and (d) show that the dot height is 1 uc of LTO by comparing the dot height with the STO substrate step height. This is consistent with the RHEED oscillation data that indicate layer by layer growth. The average dot distances were measured from the STM images. Fig.4(e) shows dot distance histograms measured from several STM images with dot coverages of ~15% and ~40 %. Based on the dot distance analysis from this histogram, the critical dot spacing appears to correspond to a charge transfer length of ~1 nm. This value is comparable to the EELS data. [1]

#### Dot size control

The results of Fig. 4 are not sufficient for percolation analysis because the dot size is dependent on coverage. Experimentally, the dot size depends on the time lag after deposition due to rapid surface migration of adatoms. Nucleation occurs during the high supersaturation period after a laser pulse. Surface migration and dot growth occurs during the period between pulses and after deposition. The time dependence of dot size was measured with the aim of obtaining surfaces with different coverage but constant dot size.

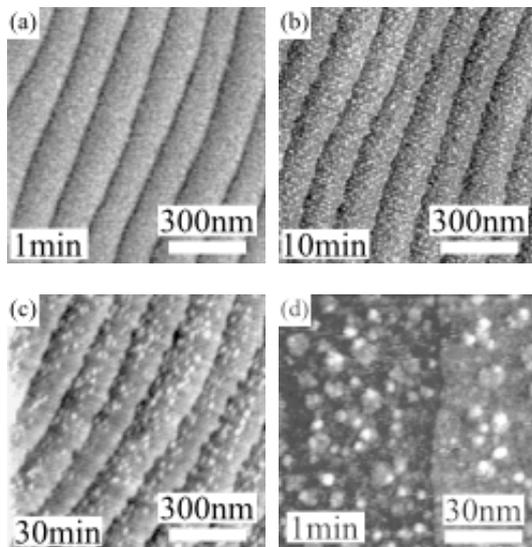


Fig.5 AFM images of surfaces annealed at 800°C for

(a) 1 min, (b) 10 min, (c) 30 min. (d) Small-area STM image of (a).

In this measurement, LTO was deposited at 500°C and annealed at 800°C for different periods of time (1min, 10min, 30min) under fixed coverage. This process aimed at eliminating the effects of varying deposition time on the average dot size.

Fig. 5 shows AFM and STM images of a 15% coverage sample. The dot sizes were measured by AFM (a)-(c). Due to a limited resolution of the AFM, it is difficult to measure the size of the smaller dots. However, STM shows that small dots were present (d). The dot diameters varied from 5nm (d) to 31nm (c). Step edge meandering can be observed in (c), indicating that many adatoms attached to step edges. For a 39% coverage sample (Not shown here) the dot diameter increased from 23 nm (1 min annealing) to 34 nm (30 min anneal), estimated from AFM images.

#### References

- [1] A.Ohtomo *et al.*, Nature **419**, 378 (2002).
- [2] A.Ohtomo *et al.*, Appl. Phys. Lett. **80**, 3922 (2002).

#### Conferences

- JSAP 72th Autumn meeting 2011[301-ZK-6], “Transport measurement of LaTiO<sub>3</sub> nanodot arrays in SrTiO<sub>3</sub>” (oral presentation), K.Sasamura, R.Takahashi and M.Lippmaa
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