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Electrical properties of Molecular Beam Epitaxy grown Barium Titanate probed by conductive Atomic Force Microscopy

Simon Martin, Nicolas Baboux, David Albertini, and Brice Gautier*

Institut des nanotechnologies de Lyon (INL), Institut National des Sciences Appliquées de Lyon, Université de Lyon, UMR CNRS 5270, 7 avenue Capelle F-69621 Villeurbanne Cedex, France

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The electrical properties of Barium Titanate 60 nm thick layers grown by Molecular Beam Epitaxy has been probed at the nanoscale by Conductive-Atomic Force Microscopy in Ultra-High Vacuum and in air, and the results compared to electrical measurements obtained on large electrodes. The role of the atmosphere of measurement is clearly highlighted by the absence of resistive memory effect when the measurements are operated in air. Considering the possibly high concentration of oxygen vacancies in such samples, we conclude that the difference might be explained by the annihilation of the oxygen vacancies by the oxygen of the environment. This emphasises the need to control precisely the atmosphere of measurement when probing the electrical properties of such thin dielectric films by atomic force microscope.

I. INTRODUCTION

Ferroelectric barium titanate $BaTiO_3$ (BTO) is considered as a good candidate for a large panel of applications ranging from electro-optic devices based on Pockels effect to low consumption transistors. Its growth by Molecular Beam Epitaxy (MBE) is encouraged in order to get a perfect epitaxy and very small thicknesses on large samples. However, one of the drawbacks of this particular growth method is the strong oxygen deficiency of the resulting layers [1]. The measurement of the electrical properties of MBE-grown BTO, e.g. the remanent ferroelectric polarization, is thus severely impinged by the presence of very high leakage currents arising from the large concentration of oxygen vacancies. The integration of such material in the devices of the micro and nano-electronics may also be delicate for the same reason.

Moreover, resistive switching effects has been reported in BTO layers, which have been linked to the direction of polarisation [2, 3] by using Piezoresponse Force Microscopy (PFM, based on Atomic Force Microscopy - AFM) images and hysteresis loops. However, the presence of oxygen vacancies, which play a non negligible role on the resistive memory effect, is a severe problem for the reliability of PFM images since they are at the origin of a parasitic signal due to the motion of vacancies under the electric field and subsequent variation of the molar volume of the material under the AFM tip [4, 5].

As a consequence, electrical properties of such layers have to be assessed carefully, so that the interpretation of PFM images does not suffer from any ambiguity, and so that ferroelectricity is linked to memory effect in a reliable way. Moreover, before these layers are considered to be integrated in electronic devices, the measurement of their true, i.e. *intrinsic* electrical properties is of first importance.

In this paper, we probe the electrical properties of BTO

thin layers and compare large scale (operated on metallic electrodes of several hundreds of micrometers size) and nanoscale (obtained with the metallic AFM tip as a top electrode) electrical measurements. We show that the results of the Current - Voltage (I-V) measurements are not the same under different environments when a positive voltage is applied to the sample (tip grounded). We propose an explanation of this phenomenon based on the presence or the absence of oxygen in the environment of measurement.

II. EXPERIMENTAL

60 nm thick $BaTiO_3$ layers have been grown by Molecular Beam Epitaxy on a $SrTiO_3$ buffer layer doped with Niobium (resistivity $\rho = 7 \text{ m}\Omega.\text{cm}^{-1}$). During the growth, a low pressure flux of oxygen was present (pressure 5.10^{-7} mbar). In these conditions the low oxygen pressure favours the presence of oxygen vacancies ([1, 6, 7]). To try and heal the vacancies, a post-deposition annealing is operated at 450°C in ambient air during one hour. High-energy electron diffraction (RHEED) spectra (not shown) show intense and well contrasted stripes indicating a good crystalline quality of the film and a flat surface. X-Ray diffraction (XRD) spectra (not shown) indicate that the films are tetragonal and c-oriented with a $c=0.408$ nm lattice parameter.

AFM measurements in air were operated with a Bruker Dimension 3100 apparatus with a PtIr5-coated tip (stiffness $\simeq 2\text{-}10 \text{ N.m}^{-1}$). In Ultra-High Vacuum (UHV), a variable temperature Omicron XA-VT apparatus was used with a pressure of $\simeq 10^{-10}$ mbar and the same kind of tips.

For I-V measurements on large electrodes, platinum electrodes of different size (from $2 \mu\text{m}$ up to $200 \mu\text{m}$) are deposited by sputtering. Current-Voltage measurements were operated with an Agilent 4156B apparatus. Here, the sample's bottom electrode is grounded while the voltage is applied through the tip, as it is the case for AFM measurements in UHV. However, during AFM

* brice.gautier@insa-lyon.fr

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