Transmission electron microscopy studies of ferroelectric ZrO2 thin films

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INTRODUCTION

The discovery of ferroelectricity in zirconia and hafnia [1] based thin films revolutionized the research in the field of ferroelectrics and paved the way for their integration into real applications. However, the wake-up phenomenon associated with the presence of non-polar phases and/or defects in the films still hinders the reliability of the device operation. Therefore, new strategies for achieving ferroelectric single-phase orthorhombic zirconia and hafnia based thin films are welcomed.

EXPERIMENTAL/THEORETICAL STUDY

8-nm thick ZrO2 thin films were grown by ion-beam sputter deposition (IBSD) onto 0.7 wt% Nb-doped SrTiO3 substrates with (001), (011) and (111) orientations.
Cross-sectional TEM samples were made by first mechanical thinning followed by Ar+ ion milling on a Gatan PIPS machine at 4 kV acceleration voltage and 6 degrees beam incidence angle. TEM observations were performed using a probe-corrected analytical high-resolution JEMARM 200F electron microscope operated at 200 kV.

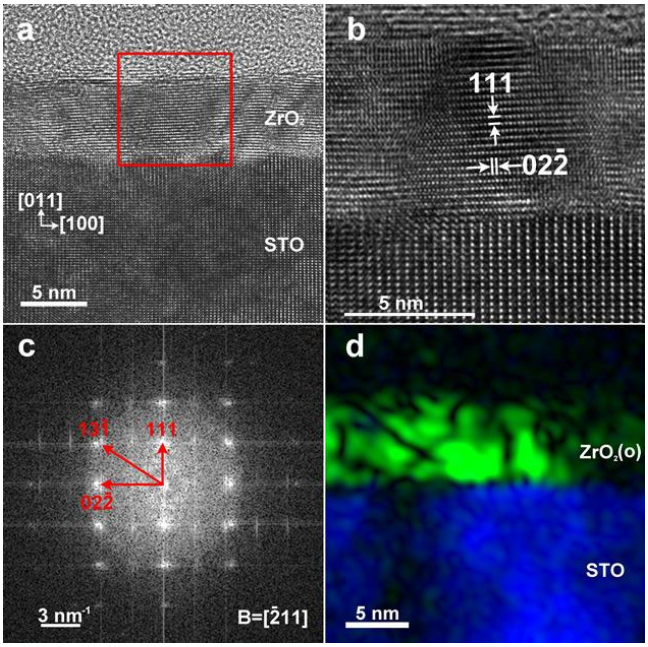
RESULTS AND DISCUSSIONS

We note the Figure inserted in the graphic area by Figure 1. Fig. 1(a), (b) show HRTEM images of a ZrO2/(011)Nb:STO thin film. The ZrO2/(011)Nb:STO structure with (111) and planes of the o-phase are indexed. The FFT pattern shown in Fig. 1(c) corresponds to an area delimited by the red square inside the ZrO2 layer in Fig. 1(a). It contains a well-defined pattern of spots, proving a high crystalline quality of the ZrO2 film in terms of grain size and preferential crystallographic orientation. The main FFT peaks were measured, indexed and assigned to the (13-1), (111) and (02-2) planes of the orthorhombic structure of ZrO2 in the [-211] zone axis orientation. [2]

CONCLUSIONS

The HRTEM investigations indicate that substrate orientation control proves to be an efficient way to manipulate the phase of the as-grown ZrO2 thin films. We also studied ZrO2 films grown on (001)Nb:STO and these second set of films showing mixed orthorhombic and monoclinic phases. Macroscopic ferroelectric measurements of the phase pure, orthorhombic (111)ZrO2 films showed a remnant polarization of ~14 μC/cm2 and a coercive field of ~1.4 MV/cm. Also, piezo force microscopy measurements showed that polar domains could be written/read and reversibly switched with a phase change of 180°.

Graphic:



Keywords:

HRTEM, Ferroelectric, Thin film

Reference:

1. T. S. Boescke, J. Mueller, D. Braeuhaus, U. Schroeder, and U. Boettger, Appl. Phys. Lett. 99, 102903 (2011)
2. José P.B. Silva, Marian C. Istrate, Markus Hellenbrand, Atif Jan, Maximilian T. Becker, Joanna Symonowicz, Fábio G. Figueiras, Veniero Lenzi, Megan O. Hill, Corneliu Ghica, Konstantin N. Romanyuk, Maria J.M. Gomes, Giuliana Di Martino, Luís Marques, Judith L. MacManus-Driscoll,
Ferroelectricity and negative piezoelectric coefficient in orthorhombic phase pure ZrO2 thin films, Applied Materials Today, Volume 30, 2023, 101708, ISSN 2352-9407,