CrystEngComm

www.rsc.org/crystengcomm

ELECTRONIC SUPPLEMENTARY INFORMATION (ESI)

Lattice guiding for sputter deposition of single domain $(Sr_{0.6}Ba_{0.4})Nb_2O_6$ ferroelectric thin films

Madhu Bhaskaran,*[a] Sharath Sriram,*[a] Brian J. Rodriguez,[b] Geethaka C. Devendra,[a] Kay Latham,[c] Dougal G. McCulloch[c] and Arnan Mitchell[a,d]

Experimental details

Thin film deposition

Strontium barium niobate (SBN60) thin films were deposited by RF magnetron sputtering from a $(Sr_{0.6}Ba_{0.4})Nb_2O_6$ target. Strontium titanate ($SrTiO_3$, STO) and 0.5 wt% niobium-doped STO (Nb:STO) substrates (10 mm × 10 mm × 0.5 mm) were utilized. The substrates were cleaned using acetone and isopropyl alcohol prior to the deposition. The conditions used for deposition are summarised in Table S1.

Table S1 SBN60 thin film deposition conditions.

Target	$(Sr_{0.6}Ba_{0.4})Nb_2O_6$
Target diameter	100 mm
RF power	200 W
Process gas	Argon
Base pressure	1.0×10^{-7} Torr
Sputtering pressure	5.0×10^{-3} Torr
Substrate temperature	700 ºC
Temperature ramp-up rate	10 ºC min^{-1}
Temperature ramp-down rate	5 ºC min^{-1}

X-ray photoelectron spectroscopy

X-ray photoelectron spectroscopy (XPS) was carried out using a Thermo Scientific K-Alpha spectrometer. The surface composition analysis was performed over a ~1 mm^2 area with rotation using an aluminium *K*α X-ray source. Carbon peaks on the substrate surface were used to verify calibration. The composition of the film was determined utilising the area under the curves, fitted and corrected with sensitivity factors using the Avantage data system. Depth profiling was performed by successive, repeated argon-ion etch and spectrometry processes.

X-ray diffraction

X-ray diffraction was performed using a Bruker D8 ADVANCE system. The radiation used was Cu $K\alpha_1$ (wavelength of 0.154056 nm). The scan was performed at room temperature for a 2θ range of 5-90º, with a step size of 0.02º and a collection time of 2 s at each step.

Transmission electron microscopy

Cross-sectional transmission electron microscope (TEM) samples were prepared by mechanically polishing using a tripod polisher to create wedge-shaped specimens, with final stages of polishing performed on a 1 μm diamond lapping sheet. The specimens were then ion milled to electron transparency at room temperature. It should be noted that preparation of electron transparent specimens for SBN60-STO structure is challenging given the brittle nature of both the film and the substrate. The TEM analysis was carried out at an accelerating voltage of 200 kV on a JEOL 2010 TEM.

Prism coupling

Optical characterisation was performed using a Metricon 2010/M prism coupler. The analysis was carried out at 532 and 633 nm wavelengths using a prism of index 2.865 and effective modal index range of 1.55-2.45 at 633 nm (Metricon 200-P-2).

Piezoresponse force microscopy

Piezoresponse force microscopy (PFM) imaging was performed using the dual-ac resonance tracking PFM mode of an Asylum Research MFP-3D atomic force microscope (AFM).[S1] PFM imaging was carried out using conducting PPP-EFM AFM tips from Nanosensors with platinum iridium coatings and nominal stiffnesses and resonant frequencies of 2.8 N m^{-1} and 75 kHz, respectively. The ac bias imaging voltages were chosen to be 0.5-1.0 V and the excitation frequencies were separated by 5 kHz and centred on the contact resonance frequency (~355 kHz). The polarization pattern was fabricated by applying +6 V *via* the tip to an 8 × 8 μm^2 area, -6 V to a 4 × 4 μm^2 area, and ±6 V (alternating) to a 2 × 2 μm^2 area.

Supplementary results

X-ray photoelectron spectroscopy

XPS depth profile results for ~175 nm thick films (1 h deposition) are presented in Fig. S1. The *x*-axis in Fig. 1 was converted from etch time (s) to etch depth (nm) by determining the etch rate (nm s^{-1}) of SBN60 during the profiling process.

Uniform composition through thickness of the film can be observed. The composition values determined are very close to those expected for SBN60, with Sr at ~6%, Ba at ~4%, Nb at ~23%, and O at ~67%.

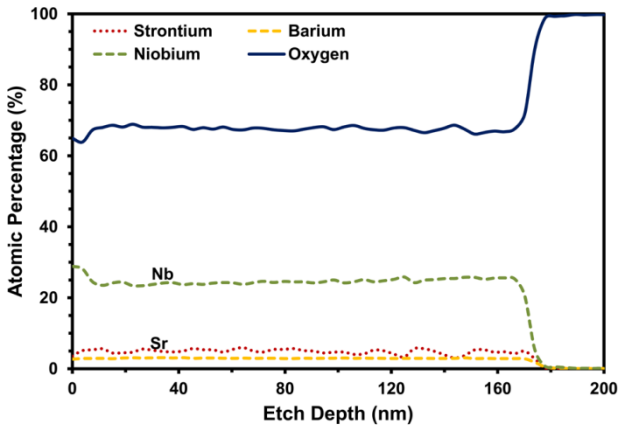

Fig. S1 X-ray photoelectron spectroscopy depth profile of ~175 nm thick SBN60 film, as-deposited under conditions in Table S1.

Selected area electron diffraction

Detailed selected area electron diffraction of the SBN60-STO interface was performed. The raw interface diffraction pattern is presented in Fig. S2. This pattern has contributions from both the film and the substrate. An indexed version is presented in Fig. 3.

Fig. S2 Selected area electron diffraction pattern from the SBN60-STO film-substrate interface. The indexed versions of this as-collected pattern are presented in Fig. 3.

Atomic force microscopy

A topography scan of the SBN60 thin film surface is presented in Fig. S3. This scan over a 3 μm × 3 μm area revealed that the root mean square roughness (R_{RMS}) of the SBN60 surface was 126 pm. This indicates that the films are as smooth as the polished substrates.

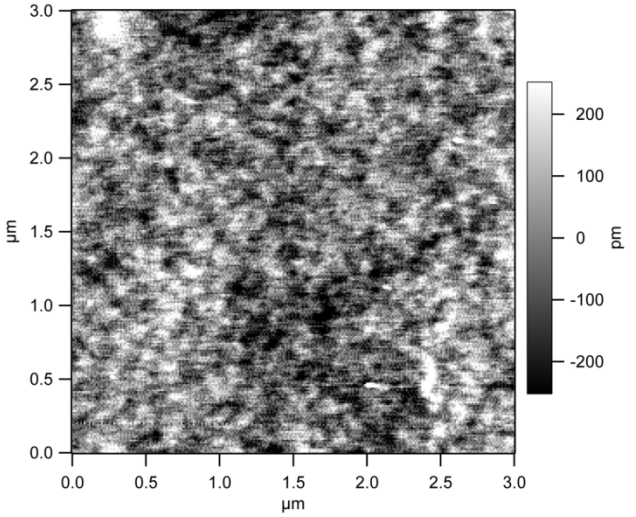

Fig. S3 Atomic force microscopy surface scan of the SBN60 thin film surface over a 3 μm × 3 μm area.

Notes and references

[a] *Microplatforms Research Group, School of Electrical and Computer Engineering, RMIT University, GPO Box 2476, Melbourne, Victoria 3001, Australia. E-mail: madhu.bhaskaran@gmail.com, sharath.sriram@gmail.com*
[b] *Conway Institute of Biomolecular and Biomedical Research, University College Dublin, Belfield, Dublin 4, Ireland*
[c] *School of Applied Sciences, RMIT University, GPO Box 2476, Melbourne, Victoria 3001, Australia*
[d] *ARC Centre for Ultrahigh bandwidth Devices for Optical Systems, RMIT University, GPO Box 2476, Melbourne, Victoria 3001, Australia*

S1 B. J. Rodriguez, C. Callahan, S. V. Kalinin and R. Proksch, *Nanotechnol.*, 2007, **18**, 475504.