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Piezoresponse Force Microscopy Study of Ferroelectric $BaTiO_3$ Thin Film Directly Deposited on Si(001) by Magnetron Sputtering

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Abstract. Direct integration of ferroelectrics with semiconductors is critical to lower the cost and simplify the production procedures for data storage/processing components and miniature sensor/actuator development. By optimizing magnetron sputtering parameters, highly <001> preferential growth of $BaTiO_3$ thin films with reproducible ferroelectric responses have been achieved on Si(001) substrates. The thin film ferroelectric characteristics were systematically studied by piezoresponse force microscopy, and a piezoelectric coefficient d_{33} of 24pm/V has been measured. It is found that the scanning tip sidewall angle and cantilever tilt affect the contour and size of polarized area.

Introduction

Ferroelectric materials with their high dielectric constants and significant peiezo/ferro-electric responses, when integrated with semiconductor substrates, can lead to the development of a wide range of functional devices. Certain ferroelectrics have now become the candidates for next generation dielectric layers in field effect transistors or capacitive components [1-4]. To develop multifunctional electro-mechanical coupled devices, it is also very desirable to develop compatible piezo/ferro-electric thin film based sensors and actuators with semiconductors [5-7]. Recently, it was found that the tunneling electrical resistance through ultra thin ferroelectric films strongly depends on their polarization direction, which makes this structure a promising type of non-destructive random access memories [8]. However, direct growth of high quality ferroelectrics on semiconductor substrates with reproducible properties has been proved to be challenging due to the thermodynamic instability and structural incompatibility of the two types of materials [9, 10]. Introducing of buffer layers can improve ferroelectric thin film growth, but it not only complicates the fabrication procedures but also can diminish the coupling effects between the ferroelectrics and semiconductors.

In this study, $BaTiO_3$ (BTO) thin films were deposited on Si substrates by radio frequency (RF) magnetron sputtering. Compared to other ferroelectric thin films processing methods such as sol-gel [11], chemical vapor deposition [12], and pulsed laser deposition [13, 14], magnetron sputtering [15-18] provides the advantages of high throughput, high growth rate, large area uniformity and compatibility with other synthesis methods. In our study, sputter deposition of BTO thin films on (001) Si substrates were optimized in terms of crystalline structures and piezoelectric responses. Thin film polarization and piezoelectric study were performed using piezoresponse force microscopy (PFM). PFM provides the capability of investigating small-scale piezoelectric properties of ferroelectric thin films. By applying an AC voltage between a conductive tip of atomic force microscope (AFM) and a conductive electrode beneath a thin film, the ferroelectric layer will be driven to deform at the frequency of applied AC voltage based on the inverse piezoelectric effect. This sample deformation will result in the generation of an AC electric signal in the AFM measured by a lock-in amplifier. The AC signal amplitude reveals the piezoelectric coefficient and the phase includes information of polarization direction [19]. PFM has been extensively used to investigate nanoscale ferroelectric domain distribution, domain boundary width [20] and domain dynamics [21]. With its additional capability to manipulate local polarization, PFM plays an important role in the development of integrate ferroelectric-semiconductor structures. On the other hand, being a developing thin film nanoscale characterization technique, there are continuous improvements in SPM technique, resolution and quantification capability [22].

In this study, piezoelectric properties of sputter deposited BTO thin films were characterized by PFM. It is observed that the PFM tip sidewall angle can influence the polarization pattern formation due to the asymmetric electrical field formation during scanning.

Experimental

BTO thin films were prepared using RF magnetron sputtering. A $BaTiO_3$ ceramic target with a purity of 99.9% was used. The base pressure of sputtering chamber was better than 4×10^{-7} Torr. As expected, film quality in terms of crystalline structure and ferroelectric response is very sensitive to sputtering parameters, the optimized growth condition was determined to be at a working gas pressure of 8×10^{-2}Torr with a Ar to O_2 ratio of 80% to 20%. The Si substrate was maintained at 650°C during deposition. The substrate heating and cooling rates were 5° C/min and 15° C/min, respectively. The target-substrate distance was 10cm and a power of 50 watt was used. Under these experimental conditions, the deposition rate was determined to be 0.25 Å/s.

X-ray diffraction experiments have been performed both on a lab x-ray diffractometer and using X-ray synchrotron diffraction at Advanced Photon Source in Argonne National Laboratory. PFM study was conducted in on a Veeco Dimension 3100 Atomic Force Microscope with Nanoscope IV controller combined with an external SR850 lock-in amplifier. The conductive tips used in PFM measurement were silicon tips coated with a 15nm Pt. Cantilever has a nominal spring constant of 45N/m and the tip radius is about 25nm.

Results and Discussion

The XRD results from a BTO thin film on Si(001) substrate taken from the laboratory x-ray diffractometer is shown in Fig. 1. A strong (002) BTO diffraction peak can be observed together with the strong (004) diffraction peak from Si substrate. Off axis synchrotron X-ray micro-beam diffraction experiments have been performed on the 2-ID-D beamline at the Advanced Photon Source in Argonne National Laboratory. The energy of the photons was selected to be 10.4 keV. The beam size used in this experiment was $6.0\times6.0\ \mu m^2$. Detailed synchrotron experiment setup can be found in Ref. [23]. Scanning of the x-ray incident beam on the sample surface results in an almost identical diffraction pattern, as shown in the inset of Fig. 1. A very bright spot can be observed on the {002} diffraction ring while the {111} diffraction exhibits as weak but uniform ring pattern. This confirms that the studied BTO sample has a very strong (001) preferential orientation perpendicular to the (001) Si substrate. Since the spontaneous polarization of BTO is also along the crystalline [001] orientations, the (001) growth of film is very desirable in application. Using small angle x-ray reflectivity measurement, BTO film thickness and surface/interface roughness were evaluated. Only thin films prepared under optimized conditions exhibits well defined multiple reflectivity peaks (inset of Fig. 1), for this sample thin film thickness is determined to be 25nm with an average surface roughness of about 2nm.

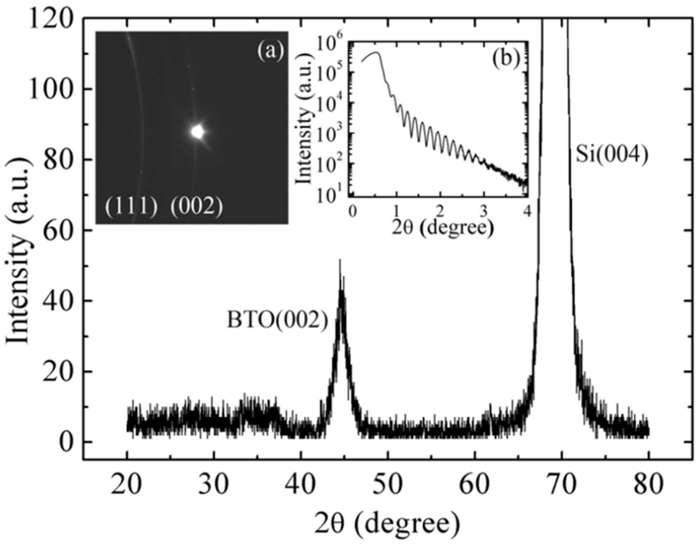

Fig. 1. X-ray diffraction pattern of a $BaTiO_3$ thin film grown on Si (001) substrate. The insets show the synchrotron X-ray diffraction pattern of the film and the small-angle reflectivity results. Diffraction patterns indicate a preferential <001> BTO orientation.

In order to characterize the ferroelectricity of the as-prepared sample, thin film polarization and reversal experiments using PFM were performed. Fig. 2(a) shows the schematics of the performed experiments. On the BTO surface, two polarization scans were conducted and the domain patterns were then imaged. Originally, in an area of 5μm×5μm (area A), a 12 V voltage was applied to the conductive AFM tip, while a -10 V DC voltage was applied to the sample substrate for a positive polarization scan. Then in the middle of this 5μm×5μm square, a smaller area of 2μm×2μm square (area B) was scanned with a reversed tip voltage of -12V and a substrate voltage of 10 V. Finally, PFM images were taken in an area of 10μm×10μm square (area C) covering the original two experiment regions.

In order to confirm the ferroelectricity of the BTO films, hysteresis loop measurements were performed after top silver electrodes were placed on the BTO surface using a Radiant RT66B tester. Fig. 3(a) shows the measurement results with different applied electric field for a 48.1nm thick BTO film. For the same sample, Fig. 3(b)-3(d) show the simultaneously recorded surface height, piezoresponse (PR) amplitude and PR phase images of the BTO thin film after the polarization experiments. Accordingly, linear scans across the polarized region are also shown in Fig. 3(e)-3(g). Fig. 3(c) suggests that the oppositely polarized areas have almost identical PR amplitude except for close to the boundary between the two areas. Linear scan shown in Fig. 3(f) provides direct quantitative information, indicating a drop in PR response in this 180^o domain boundary. In comparison, the PR phase image of the polarized region shows distinct contrast between area A and area B [Fig. 3(d)]. As being more clearly demonstrated from the linear scan shown in Fig. 3(g), PR phase responses are -90° and 90° for area A and area B, respectively. Considering the phase of the PFM driving voltage was -90°, it is deduced that area A corresponds to the in-phase domain, while area B is the out-of-phase domain. The surrounding area outside of region A, the un-polarized part of the sample (area C), has a much smaller PR amplitude compared to regions A and B and its PR phase responses have an average value close to 0°. This observation indicates that the net spontaneous polarization in as-prepared sample is close to 0 and in a depolarized state. The fact that this sample shows strong piezoresponse after polarizing verifies the ferroelectricity of the deposited BTO film.

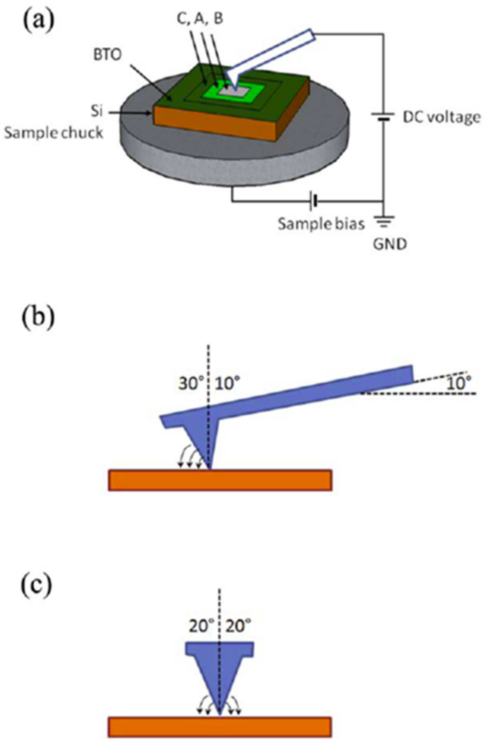

Fig. 2. (Color online) (a), Schematic illustration showing polarizing process in contact AFM. To begin with, area A (5×5 μm^2) is scanned with tip applied by a +12V DC voltage, and sample applied by a -10V DC voltage. Then area B (2×2 μm^2) is scanned with reverse voltages. After that area C is scanned by PFM. (b) and (c) are schematic illustrations showing the influence of tip sidewall angle on polarizing process.

Using PFM, the piezoelectric coefficient of the sample was also quantified. Assuming that the local electric field between the PFM tip and bottom electrode is perpendicular to the film plane and considering the (001) preferential growth of the BTO film, the piezoelectric coefficient measured by PFM should be an approximate value of d_{33}. Its value can be deduced from PR amplitude since it reflects the deformation of the BTO film after polarization. Fig. 3(f) shows the profile across the two polarized regions. In the central polarized region, an instrument voltage of about 1.2 volts was measured. Considering an amplification factor of 1000, the actual sample PR amplitude should be 1.2mV. Using the following formula, the effective piezoelectric coefficient d_{33} of the sample was calculated [24].

$$d_{33} = \frac{A_V S_d}{U}. \tag{1}$$

where A_V is the PR amplitude in unit of volts, S_d is deflection sensitivity of tip cantilever and U is drive voltage. For this study, S_d was calculated to be 99.78nm/V from the slope of force curve measurement. Driving voltage U was 5V. Therefore the effective piezoelectric coefficient d_{33} of the sample was determined to be 24pm/V. This value was much higher than the 14pm/V measured for the PPLN (periodically poled Lithium Niobate) standard in our experiment.

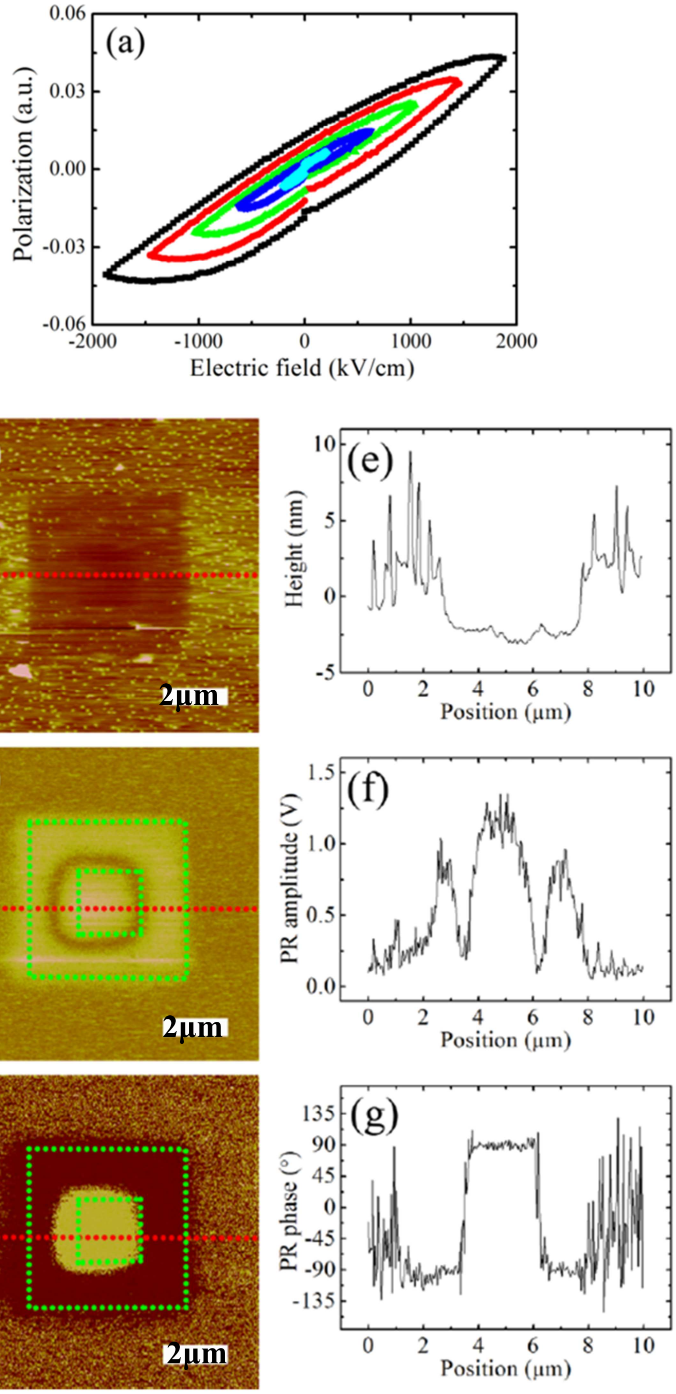

Fig. 3. (a) Ferroelectric hysteresis loops of a BTO film measured with stepping up applied electric fields, and (Color online) PFM images of a BTO thin film on Si (001) after polarizing. (b), Height; (c), PR amplitude; (d) PR phase images. (e), (f) and (g) are the profiles of the linear scan along the red dotted lines shown in (b), (c) and (d), respectively. The green dotted squares in (c) and (d) indicates the scanned areas for comparison to the polarized areas. The scale bars in (b), (c) and (d) are 2 μm.

A closer look at scanning pattern and PFM response of the BTO thin film indicated that the polarization pattern can be significantly influenced by PFM tip shape. From the surface morphology scan shown in Fig.s 3(b), we can see that there are grains with size on the order of tens of nanometers uniformly distributed on the as-deposited sample surface. These small grains were mostly removed during poling process with the use of high stiffness scanning tips to reduce electrostatic and electrostriction effect [25]. That is why the height of scanned areas (both A and B) are lower than the surrounding regions, which is clearly demonstrated in Fig. 3(d). This sample surface morphology change has been utilized to determine the exact traces of the PFM tip motion during the polarizing process.

A direct comparison between the actual PFM tip scan region [green dotted lines in Fig. 3(c) and 3(d)] and the formed polarized BTO domain pattern indicated that the two areas don't exactly overlap with each other. As shown in Fig. 3, the ferroelectric domains cannot maintain sharp edges and the domain pattern size exceeds the scanning area with apparent asymmetry. Deviation of the domain pattern from scanning area is most significant for the left edge and the least for the right edge, with top and bottom edges have comparable deviation in the middle range. Table 1 summarizes the deviations of the polarized domain along the four edges from the 5×5 μm^2 and 2×2 μm^2 scanned areas, respectively. The observed phenomenon is caused by the electrical field distribution based on tip geometry and cantilever tilt during the experiments. The conductive PFM tip used in this study has a pyramid shape with each of the four side has an angle of 40° from a middle plane. When performing PFM polarization experiments, the cantilever beam has a 10° upward tilt, as shown in Fig. 2(b), during the scan the angles between the sample plane and the four PFM tip surfaces are 60° on the left, 80° on the right [Fig. 2(b)] and 70° at top and bottom [Fig. 2(c)]. Since the angle between the PFM tip and bottom conductive electrode determines the polarization electrical field distribution, smaller angle between the tip surface and sample plane will generate large shadow area where the underlying ferroelectric thin films are polarized.

Table 1. Dimensions of polarized area beyond tip contact point when a polarizing voltage of 22V DC is used. Each value is the mean of values from PR amplitude and PR phase images in Fig. 3.

	left	bottom	Top	right
Region A (nm)	554	300	326	50
Region B (nm)	801	336	392	0

Recent studies on solid state ionic conductors and certain dielectric materials show that due to field induced electrochemical or electromechanical processes, atomic scale mechanical deformation or bulk charge transfer can provide signals similar to a piezoelectric response in scanning probe microscopy (SPM) based measurements [26-28]. In this study, AFM and PFM contrast dependence studies were performed by altering polarization field levels. It is found that there exists two opposite critical voltage levels beyond which no observable changes in AFM and PFM responses can be identified. In between these two critical voltages, contrast between the polarized and un-polarized region does depend on the magnitude of the writing electric field. This observation together with the 180° phase difference between two opposite polarization orientations demonstrated that the synthesized BTO has two ferroelectric states.

Summary

In summary, <001> preferential BTO crystalline thin films were grown on Si(001) substrates by using RF magnetron sputtering with desirable ferroelectricity. As-prepared samples can be easily polarized and polarization can be switched in local areas using PFM. A piezoelectric coefficient as large as 24pm/V have been measured for the <001> BTO. BTO microstructure and ferroelectricity is sensitive to sputtering conditions, but experimental results are reproducible once the optimized parameters are determined. Currently interfacial microstructure and BTO growth mechanism on Si are still under investigation. This study demonstrates possibility of direct integration of ferroelectric thin films with semiconductors for nanoscale device applications. SPM investigations also show that small SPM tip angle and less cantilever tilt will lead to the generation of better defined domain patterns with less asymmetry along different edges.

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