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Growth, crystal structure, and properties of epitaxial BiScO₃ thin films

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Epitaxial thin films of BiScO₃—a compound thermodynamically unstable under ambient conditions—were grown on BiFeO₃-buffered SrTiO₃ substrates. X-ray diffraction confirmed the reasonable crystalline quality of the films with a full width at half maximum of 0.58° in ω (004 reflection), 0.80° in φ (222 reflection), and 0.28° in θ. The epitaxial thin films of BiScO₃ on SrTiO₃ retain the principal structural features of bulk BiScO₃ (i.e., octahedral tilting and the pattern of Bi displacements) that give rise to a pseudo-orthorhombic unit cell 2 × 2a × 4a (a = 4 Å refers to the lattice parameter of an ideal cubic perovskite). Films grown on (100) substrates adopt the bulk monoclinic structure, whereas films on the (110) substrates appear to exhibit an orthorhombic symmetry. The dielectric permittivities were modest (<30) with low loss tangents (<1% at low fields); no maxima were observed over a temperature range of ~200 to +350 °C. There is no evidence of significant hysteresis (either ferroelectric or antiferroelectric) at room temperature up to the breakdown strength of the films. © 2008 American Institute of Physics.

INTRODUCTION

Ferroelectric lead zirconate titanate (PZT) ceramics are the basis for a large number of commercial piezoelectric devices.¹ Of particular interest are the superior properties found near the morphotropic phase boundary (MPB).² However, the relatively low Curie temperatures (Tc ~380 °C at the MPB) of the PZT system limit their utility in some applications. Moreover, the drive to move away from lead-containing piezoelectrics is spurring considerable research. In the search for new high temperature or lead-free piezoelectrics, Bi-based perovskite compounds are of interest.³ Recently, (1−x)BiScO₃−xPbTiO₃ ceramics have been reported to exhibit piezoelectric properties (d₃₃ ~460 pC/N) comparable to that of PZT and high ferroelectric transition temperature Tc=450 °C near the MPB.⁴,⁵ Both single crystals⁶ and thin films⁷,⁸ also show good properties. Thin films of other BiScO₃ solid solutions have also been reported.⁹,¹⁰

Despite its utility in ceramic solid solutions, there is little knowledge about the end member BiScO₃ itself. As a family, Bi-based ABO₃ perovskite-like oxides are attracting considerable attention because of their potentially exploitable functional properties. For example, BiFeO₃ (Ref. 12) and BiMnO₃ (Refs. 13 and 14) are multiferroics, whereas BiScO₃, as described above, is an interesting end member for both dielectric and piezoelectric solid solutions. Most of the known BiBO₃ perovskites (including BiMnO₃ and BiScO₃) are unstable under ambient conditions, and their synthesis typically requires high pressures.¹⁵,¹⁶ This is likely due to a low structural stability for the perovskite phase that originates from the rather small tolerance factor.¹⁷ The crystal structures of BiFeO₃ (Ref. 18) and BiMnO₃ (Ref. 19) have been established. Recently, the structure of BiScO₃ synthesized under high pressure was determined using neutron powder diffraction and electron diffraction.²⁰,²¹ According to these studies, BiScO₃ is isostructural with BiMnO₃ and exhibits a monoclinic C2/c symmetry with lattice parameters a=9.889 Å, b=5.822 Å, c=10.046 Å, and β=108.3°. Despite the existing knowledge of atomic positions, the origins of structural distortions (i.e., octahedral tilting, cation displacements, etc) that give rise to complex superstructures in both BiMnO₃ and BiScO₃ compounds have not been discussed. However, identifying these distortion mechanisms is essential both to an understanding of the displacive behavior in Bi-based perovskites as well as to the functional properties.²² In particular, it is not known at present whether BiScO₃ is ferroic. Although it has previously been speculated that it may be either ferroelectric or antiferroelectric, no experimental confirmation was reported.²³,²⁴ In the present work, these questions are addressed by stabilizing the perovskite phase in BiScO₃ thin films using epitaxy, thereby permitting structural and electrical characterization. The structure of thin films was compared to that of a bulk BiScO₃ sample synthesized under high pressure.²⁵

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EXPERIMENTAL PROCEDURE

BiScO₃ films, BiFeO₃ buffer layers and SrRuO₃ bottom electrodes were all prepared on (100) and (110) SrTiO₃ single crystal substrates using pulsed laser deposition (PLD). A KrF excimer laser [Lambda Physik Compex 305i (the use of brand or trade names does not imply endorsement of the product by NIST)] with a 248 nm wavelength was used to ablate the target materials. The laser energy density used for BiScO₃ growth was ≈1.7–1.8 J/cm². SrRuO₃ bottom electrodes and BiFeO₃ buffer layers were deposited using stoichiometric ceramic targets (Target Materials Inc.). Details of their epitaxial growth are given elsewhere. BiScO₃ films were deposited using a mixed phase sintered ceramic target with 5 mol % excess Bi₂O₃ to compensate for the loss of bismuth during growth. The BiScO₃ target was sintered at 825 °C for 1 h.

(100) and (110) SrTiO₃ substrates (CrysTec GmbH) were first ultrasonically cleaned sequentially in trichloroethylene, acetone, and isopropanol and then mounted to the substrate heater with Ag paint. The chamber was pumped down to a base pressure of 10⁻⁵ – 10⁻⁶ Torr and then backfilled with Ar, O₂, excess Bi₂O₃. For initial growth runs in a background of 100 mTorr O₂, excess Bi₂O₃ was no longer detected at deposition temperatures beyond 670 °C; thus, BiScO₃ was typically grown at 670 °C. As reported previously, for the growth conditions employed, phase pure perovskite BiScO₃ films could not be grown on SrTiO₃ or SrRuO₃/SrTiO₃ substrates. Instead, diffraction peaks from Bi₂O₃ and Sr₂O₃ were detected with a trace of perovskite phase. Under the same growth conditions, a thin BiFeO₃ buffer layer (5–10 nm) ameliorates this problem, as shown in Fig. 1. In this work, BiFeO₃ buffer layers of either 5 or 7.5 nm thickness were employed. The (001) orientation was obtained on (100) SrTiO₃, where the subscript c refers to an ideal cubic perovskite unit cell with a₀ = 4 Å. The out-of-plane pseudocubic lattice constant was 4.137 ± 0.001 Å, which is larger than those reported for bulk ceramics. The full width at half maximum (FWHM) of the BiScO₃ 002, and 222, diffraction peaks in the θ = 2θ and φ scans were 0.28° and 0.8°, respectively. The fourfold periodicity of the φ scan of the BiScO₃ 222 peaks demonstrates that cube on cube epitaxy was achieved.

Even with the BiFeO₃ buffer layer, it is observed that the growth rate (laser frequency) affects the film phase purity dramatically. Figure 2 shows the XRD patterns of samples that were grown at 670 °C (100 mTorr oxygen background) but with different laser frequencies. Clearly, the intermediate growth rate results in the best crystallinity and phase purity. At lower rates, a minor peak appears at ≈32°, which may come from a {110}-oriented perovskite phase or a second phase such as Sc₂O₃. The optimized growth condition was identified as Tsub=670 °C, 100 mTorr O₂ background pressure, and 10 Hz laser frequency.

BiScO₃ films were grown on {110} SrTiO₃ using the same deposition conditions. Out-of-plane XRD measurements show an excellent orientation for the BiScO₃, while the TEM measurements described below confirm epitaxy.

### Structure determination

The reported BiScO₃ structure reveals a coexistence of the two principal effects: (1) octahedral tilting and (2) antiparallel Bi displacements similar to those of Pb in PbZrO₃. The octahedra exhibit periodic antiphase tilting about two of the ⟨001⟩c directions, which results in the \( \sqrt{2}a_c \times \sqrt{2}a_c \times 2a_c \) orthorhombic superlattice. Octahedral tilting about the third ⟨001⟩ direction parallel to the c-axis of this cell is irregular, being obscured by severe distortion of [ScO₆] octahedra (Fig. 3); therefore, the overall tilting topology in BiScO₃ cannot be easily described using Glazer notation. Bi displacements, directed along the b-axis of the orthorhombic cell, double the periodicity along both a- and c-axes to yield \( 2\sqrt{2}a_c \times \sqrt{2}a_c \times 4a_c \). Hereafter, this orthorhombic cell will be denoted using a subscript o. The displacement patterns for Bi in the successive cₙ-planes are illustrated in Fig. 4. A distinct sequence of Bi cₙ-planes is supported by the oxygen displacements that take the form of periodic bending of octahedra (Fig. 3). The actual BiScO₃ structure is distorted from orthorhombic and exhibits a monoclinic symmetry with the lattice parameters \( a_{m} \approx \sqrt{6}a_{c} \), \( b_{m} \approx \sqrt{2}a_{c} \), \( c_{m} \approx 6a_{c} \), and \( β \approx 108° \). The pseudo-orthorhombic and monoclinic lattices are related according to \( a_{o}=a_{m}+c_{m}, b_{o}=b_{m}, \) and \( c_{o}=a_{m}-c_{m} \).

RESULTS AND DISCUSSION

Film growth

For initial growth runs in a background of 100 mTorr O₂, excess Bi₂O₃ was no longer detected at deposition temperatures beyond 670 °C; thus, BiScO₃ was typically grown at 670 °C. As reported previously, for the growth conditions employed, phase pure perovskite BiScO₃ films could not be grown on SrTiO₃ or SrRuO₃/SrTiO₃ substrates. Instead, diffraction peaks from Bi₂O₃ and Sc₂O₃ were detected with a trace of perovskite phase. Under the same growth conditions, a thin BiFeO₃ buffer layer (5–10 nm) ameliorates this problem, as shown in Fig. 1. In this work, BiFeO₃ buffer layers of either 5 or 7.5 nm thickness were employed. The (001) orientation was obtained on (100) SrTiO₃, where the subscript c refers to an ideal cubic perovskite unit cell with a₀ = 4 Å. The out-of-plane pseudocubic lattice constant was 4.137 ± 0.001 Å, which is larger than those reported for bulk ceramics. The full width at half maximum (FWHM) of the BiScO₃ 002, and 222, diffraction peaks in the θ = 2θ and φ scans were 0.28° and 0.8°, respectively. The fourfold periodicity of the φ scan of the BiScO₃ 222 peaks demonstrates that cube on cube epitaxy was achieved.

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BiScO₃ films were grown on {110} SrTiO₃ using the same deposition conditions. Out-of-plane XRD measurements show an excellent orientation for the BiScO₃, while the TEM measurements described below confirm epitaxy.

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The room-temperature lattice parameters of BiScO$_3$ in the orthorhombic setting are $a_o = 11.676$ Å, $b_o = 5.822$ Å, $c_o = 16.159$ Å, and $\beta = 90.95^\circ$.

As a reference for the thin films, electron diffraction patterns from a bulk BiScO$_3$ pellet were collected. BiScO$_3$ pellets were prepared by cold pressing of a powder sample at about 2.5 GPa. The synthesis of these samples is described elsewhere.$^{20}$

Selected area electron diffraction patterns (Figs. 5 and 6) for the bulk sample support the $C_{2/c}$ monoclinic structure deduced previously from x-ray and neutron powder diffraction data. Lack of the regular in-phase octahedral tilting is evident from the absence of reflections. Individual grains in this sample contained multiple rotational (twin-type) variants of the monoclinic cell (Fig. 7). The diffraction patterns revealed nearly continuous rods of diffuse intensity parallel to the $c_o$-axis of the pseudo-orthorhombic cell, i.e., $h0l_{m}$ reflections. Dark-field imaging (Fig. 7) confirmed that these rods are associated with a high incidence of planar defects, which can be attributed to the antiphase shifts for the directions of Bi displacements in successive $c_o$-planes. Nonzero intensities at $k = \frac{1}{2}[110]_c$ (asterisk indicates reciprocal space) presumably arise from the intersection of these rods with the Ewald sphere (Fig. 6), as confirmed by tilting the crystals around both $a_o$ or $b_o$ axes. Some additional disorder in the directions of Bi displacements within the $c_o$-planes is reflected in the diffuse streaking along the $[101]_m/[100]_c$ direction.

The reciprocal lattices of both $|001|_c$ and $|110|_c$-oriented films, reconstructed from the series of electron diffraction patterns, were consistent with the pseudo-

FIG. 1. XRD for BiScO$_3$ films grown on (a) SrRuO$_3$/SrTiO$_3$ (100) and (b) BiFeO$_3$/SrRuO$_3$/SrTiO$_3$ (100) substrates. The depositions were done at a substrate temperature of 670 °C, an oxygen pressure of 100 mTorr, and a laser frequency of 10 Hz. (c) $\phi$ scan of the BiScO$_3$ 222 reflection for the same film shown in (b). Peaks with an asterisk are from the SrTiO$_3$ substrate.

FIG. 2. (Color online) XRD of BiScO$_3$ films grown on BiFeO$_3$/SrRuO$_3$/SrTiO$_3$ (100) substrates at a substrate temperature of 670 °C and an oxygen pressure of 100 mTorr. The laser frequency (growth rate) dramatically changes the film phase and crystallinity. The reflections are indexed according to the pseudocubic unit cell.

FIG. 3. A projection of the octahedral framework in the BiScO$_3$ structure along the [101]$_c$ direction of the $C_{2/c}$ cell (parallel to the c-axis of the orthorhombic cell $2a_c \times 2a_c \times 4a_c$). Octahedral tilting about the viewing direction is very irregular due to a pronounced distortion of the $[ScO_6]$ octahedra.
with the variants with parallel ing arrows in the monoclinic and pseudo-orthorhombic cells. The diffuse spots indicated us-
using solid and dotted arrows, respectively.

clearly observed in the orthogonal co intersection of such diffuse relrods from another twin variant relrods parallel to
encircled using a dotted line in the sive co

FIG. 6. Schematic representation of the reciprocal lattice for a single do-

FIG. 7. Diffraction contrast images of individual grains in the bulk BiScO3 sample. (a) Bright-field image reveals twin domains representing variants of the pseudo-orthorhombic cell with orthogonal c-axes. (b) Dark-field image recorded with the 110m reflections highlights the high density of planar interfaces on the (001),⟨⟨202⟩⟩ planes that represent antiphase and monoclinic twin-domain boundaries.

orthorhombic cell of the bulk BiScO3, 2√2a×√2a×4a, (Fig. 8). However, the domain structures and details of structural distortions varied markedly with the orientation of SrTiO3, as described below.

BiScO3 films grown on {100} SrTiO3 yield electron diffraction patterns similar to those from the bulk samples (Fig. 9). The films consist of four twin-related variants of the orthorhombic structure (Fig. 10). For each domain, the a'- and b'-axes are parallel to the out-of-plane ⟨101⟩ directions, while the c'-axis (4a) is parallel to the in-plane ⟨100⟩ direction (Fig. 8). No domains with the out-of-plane c'-axis were observed. Clear and reproducible intensity variations among the 111o and 111m reflections are consistent with the monoclinic symmetry adopted by the bulk structure. Similar to the bulk samples, films on {100} SrTiO3 exhibit a high incidence of planar interfaces (monoclinic twin domain boundaries and antiphase defects) that generate streaks of diffuse intensity along the c'-axis.

In contrast to the films on {100} substrates, the BiScO3 films on {110} SrTiO3 feature a single orthorhombic variant having a'- and c'-axes parallel to the out-of-plane ⟨110⟩o and in-plane ⟨100⟩o directions, respectively (Figs. 8 and 11). For these films (Fig. 11), no obvious signatures of monoclinic distortion were observed (note the similar intensity of the 111o and 111m reflections). Similar to the bulk samples and films, the relrods of diffuse intensity pass through the super-lattice reflections parallel to the c'-axis. The principal differ-

FIG. 6. Schematic representation of the reciprocal lattice for a single do-

FIG. 8. Schematic representation of the orientation relations between the BiScO3 film and the (a) ⟨100⟩ and (b) ⟨110⟩ SrTiO3 substrates. In (a), only one (out of four) rotational variants of BiScO3 is indicated for clarity.
ence between the reciprocal lattices of the \{110\}_c and \{100\}_c films is associated with the presence of strong sharp \(hhl_{2n}\) and weak diffuse \(hhl_{2n+1}\) reflections in the former (the opposite is observed for the \{100\}_c films), which suggests a dissimilar symmetry of the two types of films. These differences in the symmetry can be attributed to distinct stacking sequences of the Bi planes along the \(c_p\)-axis. Evidently, both types of films, as well as the bulk structure, exhibit a pronounced tendency toward the formation of the \(c_p\)-plane stacking defects as reflected in the presence of the \(c_p\)-axis diffuse relrods in all BiScO\(_3\) samples. Assuming Bi displacements along the \(b\)-axis, as in the bulk structure, Bi displacements in the BiScO\(_3\) thin films are directed out of the film plane, regardless of substrate orientation.

**Electrical and optical characterization**

Spectroscopic ellipsometry was used to analyze the depth profile of typical samples. Reference dielectric function data were first obtained for BiFeO\(_3\) from thin film BiFeO\(_3\) samples grown on SrTiO\(_3\). These data were then used to model a BiScO\(_3\)/BiFeO\(_3\)/SrTiO\(_3\) stack. It was found that over the wavelength range from 350 to 650 nm, the optical properties of BiScO\(_3\) could be well described by

\[
\varepsilon = 1 + \frac{A\lambda^2}{\lambda^2 - \lambda_o^2},
\]

where \(\lambda\) is the wavelength in nanometers, \(\varepsilon\) is the dielectric function, \(A=3.81 \pm 0.15\), and \(\lambda_o=237.1 \pm 8.8\) nm. No improvement in the fit to the data was obtained by adding...
damping to the oscillator. It was also found that the films were extremely smooth, with well-defined interfaces; the modeled thicknesses for the BiFeO$_3$ and BiScO$_3$ were within experimental error of measurements made by other techniques.

Figure 13 shows the capacitance and loss tangent between $-200$ and $400 \, ^°C$ for a Pt/BiScO$_3$/BiFeO$_3$/SrRuO$_3$/[100] SrTiO$_3$ sample. There is little dispersion in the dielectric constant, and the dielectric loss was low over the entire measurement range, suggesting that BiScO$_3$ is a good electrical insulator to at least $400 \, ^°C$. The capacitance data, coupled with an estimated BiFeO$_3$ permittivity along $\langle 001 \rangle$, of $70$ was used to estimate the BiScO$_3$ permittivity. It was found for films on $\{100\}$ substrates that the BiScO$_3$ films had permittivity measured through the film thickness between 22 and 36 (for 80 nm thick films). The calculated values lie between 21 and 34 if the BiFeO$_3$ is assumed to be conducting. This value is approximately half of that of the reported value for ceramic samples. A 245 nm thick film deposited on $\{110\}$ SrTiO$_3$ had a room-temperature dielectric constant of approximately 57 (calculated using a zero field permittivity for the BiFeO$_3$ film along $\{110\}$, of 90). The slight increase in the dielectric constant with increasing temperature could arise either from a possible phase transition at temperatures beyond 400 $^°C$ or potentially from the BiFeO$_3$. However, the smooth increase in permittivity with temperature suggests that there are no ferroic phase transitions in the BiScO$_3$ between $-200$ and $+350 \, ^°C$.

Higher electric fields were applied both on samples with top and bottom electrodes and on samples with interdigitated top electrodes only in an attempt to investigate whether the samples show any characteristic switchable polarization. As can be seen in Fig. 14, there was no detectable ferroelectric or antiferroelectric behavior that developed out of plane at electric fields up to 550 kV/cm for samples on $\{100\}$ SrTiO$_3$. Instead the sample acted as a linear capacitor with a small dielectric loss at room temperature to the available range of field levels. The slope of the curve was consistent with the low field permittivity values. At 60 $^°C$, there was no significant difference in the shape of the curves, except for an increase in the loss. Similarly, measurements made in plane on films on $\{100\}$ SrTiO$_3$ showed no clear ferroelectric or antiferroelectric characteristics at room temperature (although in this case, only 50 kV/cm could be applied and the electric field will be poorly confined in the BiScO$_3$ layer).

To conclude, epitaxial BiScO$_3$ films were grown on BiFeO$_3$-buffered SrRuO$_3$/SrTiO$_3$. The results suggest that the epitaxial thin films of BiScO$_3$ on SrTiO$_3$ retain the principal structural features of the bulk ceramics synthesized at high pressures (i.e., octahedral tilting and patterns of Bi displacements) that give rise to a pseudo-orthorhombic unit cell $2\sqrt{2} a_x \times 2 a_c \times 4 a_c$. However, substrate orientation modifies details of the structural distortion along with the types and configurations of the twin domains: films on $\{100\}$ SrTiO$_3$ adopt the bulk monoclinic structure, whereas the films on $\{110\}$ SrTiO$_3$ appear to exhibit an orthorhombic symmetry. Similar to the bulk structure, all BiScO$_3$ films exhibit a pronounced disorder of Bi displacements. No phase transitions were apparent in the dielectric data between $-200$ and $+350 \, ^°C$. The effective permittivity for a stack of 10 nm of BiScO$_3$ on 5 nm of BiFeO$_3$ was $35 \pm 3$.

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