Multiferroic perovskites under epitaxial strain
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Chapter 3

TbMnO$_3$ thin films on (001)-SrTiO$_3$: growth and structure.

3.1 Abstract

In this chapter, we present the growth and structural characterization of TbMnO$_3$ thin films on (001)-oriented SrTiO$_3$ single crystals. (001)-SrTiO$_3$ is preferred in studies of epitaxy in perovskites because it can be obtained with atomically flat surfaces, favoring high quality growth. The films are shown to be very flat, c-oriented and compressively strained. Their unit cell is orthorhombic with an orthorhombic distortion that can be tuned with the film thickness up to a thickness of about 50nm. A fine domain microstructure has been revealed by synchrotron x-ray diffraction and transmission electron microscopy, showing an increasing number of orthorhombic domains as the thickness is decreased. Additionally, we show that the density of domain walls follows a linear dependence with the inverse of thickness.

3.2 Substrate treatment

Prior to deposition, the single crystals of SrTiO$_3$ (with size 5x5x0.5mm$^3$) were chemically and thermally treated in order to obtain high quality surfaces, following the procedure reported in reference [98]. Figure 3.1(a) shows the AFM image of an as-received SrTiO$_3$ substrate. Although a steps and terraces structure is clearly visible and the ter-
Figure 3.1: (a) Typical AFM topography image of an as-received (001)-oriented SrTiO$_3$ substrate. (b) Procedure elaborated by ref. [98] to obtain high quality substrate surfaces. (c) AFM topography image for a TiO$_2$-terminated (001)-SrTiO$_3$ substrate. (d) Profile along the line seen in (c). The 4Å steps are clearly visible and the flatness of the terraces is evidenced.

As shown in figure 3.1(b), the treatment procedure includes, first, the cleaning of the substrates with acetone and ethanol. The substrates are then put into water so that a chemical reaction between SrO and H$_2$O can occur, forming strontium hydroxide (Sr(OH)$_2$). The strontium hydroxide is then dissolved in a commercial solution of mixed ammonium fluoride (NH$_4$F, 87.5%) and ammonium hydrogen difluoride ((NH$_4$)HF$_2$, 12.5%) during a short time of 30s, enough to dissolve the strontium hydroxide without attacking the surface of the substrate and creating holes (so called 'etch pits'). At this point, the substrates are TiO$_2$ terminated and present steps of 4 Å. The final step is to induce a thermal motion of the adatoms at the surface of the substrate to obtain straight terraces. The length of the terraces is given by the miscut
angle of the substrate which determines then the duration of the thermal treatment. This implies that the smaller the miscut, the longer the time needed for the adatoms to reach a step. However, even though the time is miscut dependent, the temperature at which the substrate are treated is constant. In our case, a temperature of 960°C was found to give good results. Figure 3.1 (c) shows an AFM topography picture of a treated substrate. The terraces are well defined and straight. Moreover, the flatness of the terraces and the height of the steps of 4 Å are evidenced by a profile line, shown in figure 3.1(d), taken along the line shown in figure 3.1(c). All the samples grown on SrTiO₃ in this thesis were deposited using substrates treated using this procedure.

### 3.3 Growth conditions

The films were grown by pulsed laser deposition, using the system described in chapter 2. A stoichiometric target of TbMnO₃ was prepared by means of a standard solid-state reaction. The films discussed in the thesis were grown at a temperature of 750°C in oxygen pressures of 0.25 mbar and 0.9 mbar. The laser energy density on the target was set at 2 J/cm², with a pulse repetition rate of 1 Hz and a distance between the surface of the substrate and the surface of the target of 55 mm. After deposition, the films were slowly cooled down (-3°C/min) to room temperature after an oxygen pressure of 100 mbar was inserted in the chamber.

### 3.4 Structure and microstructure.

About eight RHEED intensity oscillations can be seen at the beginning of the growth corresponding to an initial layer-by-layer (2D-like) growth, see figure 3.2 (a). However, those oscillations are superimposed on a decreasing intensity background, indicating that an overall roughening occurs as the growth proceeds. After the oscillations have faded away, the intensity of the specular spots remains constant and the pattern

Figure 3.2: (a) Intensity of the specular spot of the RHEED pattern as a function of deposition time. The inset is a blow-up of the first RHEED oscillation showing the laser pulses. (b) Typical RHEED pattern obtained after growth and annealing. The picture is taken after cooling down to room temperature and under vacuum. (c) and (d) AFM images of an 2nm and 8nm thin film, respectively, grown at $pO_2 = 0.25$ mbar. The vertical and horizontal scales are shown at the bottom of the pictures. (e) and (f) Profile of the surface taken along the lines drawn in (c) and (d), respectively.

changes from a purely stripy pattern to the mixed stripe-spot pattern shown in Figure 3.2(b).

Figure 3.2 (c) shows the morphology of a 2nm film grown at 0.25mbar. It can be seen that the film is atomically flat, showing the step-and-terrace morphology of the substrate. However, when the deposition is stopped after 8nm, particles with a diameter of around 30 nm start appearing, as shown in figure 3.2 (d). In figure 3.2 (e) and (f), profile lines were taken in the AFM images of the 2nm and 8nm film, respectively. The profile line for the 8nm film was chosen to pass through a particle at the surface. This shows that for a 2nm film, the features at the surface have a height corresponding to half-unit cell to 1 unit cells whereas the particles on the 8nm sample have a height corresponding to one or two unit cells, showing the beginning of the islands nucleation.

Figures 3.3(a)-(c) show AFM images of three TbMnO$_3$ films with different thicknesses grown under the same conditions. In the 17nm film (figure 3.3 (a)), the grains
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Figure 3.3: AFM images of 17nm (a), 34nm (b) and 67nm (c) thick TbMnO$_3$ thin films grown with a pO$_2$ = 0.25 mbar. The scans area is 1µmx1µm. The corresponding diffraction patterns can be seen in (d), (e) and (f), respectively. The experimental data are the thick black lines. The fit to the data is shown in each case as a thin line.

dominate the film morphology but the steps from the substrate are still visible. Figures 3.3 (d)-(f) show typical 2θ-ω x-ray diffractograms along the (00L)$_c$ crystal truncation rod (the most intense peak in the pattern corresponds to the reflection of the substrate), for the same three films. The film peaks are those at the right hand-side of the SrTiO$_3$ reflections. The grey lines are the fit to the crystal truncation rods using a program based on a kinematical model. These calculations take into account, not only the complex refractive index of the substrate and the film, but also the angular dependent atomic scattering factors [99]. Despite the island-like growth discussed previously, the high quality and flatness of the interfaces is evidenced by the presence of Laue fringes around the film peak for thicknesses up to 67nm.

Figure 3.4 shows a broader 2θ-ω scan including both the (001)$_c$ and the (002)$_c$ SrTiO$_3$ Bragg reflections$^1$. No secondary or impurity phases could be observed. A phi-scan around the (024)$_{pc}$ (i.e. (228)$_o$) of TbMnO$_3$ (see inset) shows the peaks separated 90

$^1$the subscript $c$ refers to cubic indexes whereas the subscripts $pc$ and $o$ stand for pseudo-cubic and orthorhombic, respectively.
degrees from each other, confirming the four-fold symmetry of the film and the coherent growth. Moreover, shoulders can be seen around each peak in the phi-scan. For clarity, they will be explained later on this chapter. X-ray diffraction, thus, shows that the films are single phase and (001)-oriented (with the $c$-axis perpendicular to the surface). From the position of the film peaks of figure 3.4, an out-of-plane lattice spacing of 7.474 Å, which is larger than the reported bulk value of 7.402 Å, was found. This value is also larger than that expected for the compressively strained samples, as will be discussed later.

A larger out-of-plane lattice parameter than the expected one could be explained by a film whose strain is relaxed by means of oxygen vacancies, as they are known to expand the unit cell [100, 101]. To investigate this possibility, the oxygen pressure during deposition was increased up to 0.9 mbar (the maximum attained in our set-up). Increasing the oxygen pressure during growth reduced the growth rate, as expected from the decrease of the plume size, and hampered the initial layer-by-layer growth, leading to a 3D growth associated with a loss of RHEED oscillations. However, the RHEED patterns after annealing, as well as the optimal growth parameters, such as the
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Figure 3.5: Out-of-plane lattice parameters of $\text{TbMnO}_3$ films as a function of oxygen pressure during growth, for a substrate temperature of 750°C.

Figure 3.6: Reciprocal space maps around the (103)$_c$ Bragg reflection of the SrTiO$_3$ substrate for 17nm, 34nm and 67nm films grown at 0.25 mbar. The vertical and horizontal lines are guides to the eyes. The abscissa (ordinate) represents the in-plane (out-of-plane) component of the scattering vector. Both axis are normalized by $2k_o=4\pi/\lambda$.

repetition rate or temperature are similar to the ones related to figure 3.2 (b). Within the range of oxygen pressures investigated, the out-of-plane lattice parameter indeed decreases linearly with increasing oxygen pressure during deposition, consistent with the number of oxygen vacancies decreasing for increasing pressures, as shown in figure 3.5. Luckily, the maximum pressure of 0.9mbar seems to be large enough to produce stoichiometric films since the unit cell of the relaxed layers that are grown at large thicknesses is equal to the bulk one, as it will be shown later.

A detailed investigation of the film structure was performed using x-ray diffraction

![Figure 3.7](image)

\textbf{Figure 3.7}: (a) RSM around (206)\textsubscript{o} and (026)\textsubscript{o} for a 67nm thick \textit{TbMnO}_3 film grown at 0.25 mbar. The arrow indicates the \(k_{\text{par}}\) of the \textit{SrTiO}_3 substrate. (b) RSM around the (103)\textsubscript{c} \textit{SrTiO}_3 Bragg reflection for a 80nm thick \textit{TbMnO}_3 film grown at 0.9 mbar. The abscissa (ordinate) represents the in-plane (out-of-plane) component of the scattering vector, both normalized by \(2k_o=4\pi/\lambda\).

To map selected areas of the reciprocal space. Figures 3.6 (a)-(c) show reciprocal space maps (RSMs) around the (103)\textsubscript{c} Bragg reflections of a 17nm, 34nm and 67nm thick film, respectively, grown at 0.25mbar. The peaks originating from the films (substrate) are at larger (smaller) \(K_{\text{perp}}\) component (see horizontal lines in the figures). The in-plane and out-of plane components of the (103)\textsubscript{c} and (013)\textsubscript{c} \textit{TbMnO}_3 peaks do not change as a function of thickness and the in-plane lattice parameter remains identical to that of \textit{SrTiO}_3 for thicknesses up to 67nm, indicating that the films are coherent with the substrate along the pseudo-cubic [100] or [010] directions. As the thickness is increased, only a broadening of the rocking curve of the \textit{TbMnO}_3 film can be seen. However, when the RSM is taken around the (113)\textsubscript{c}, a different scenario is observed, as shown in figure 3.7 (a) for a 67nm thin film. Instead of a coherent peak, two peaks, corresponding to the film, are found that could be indexed as orthorhombic (206)\textsubscript{o} and (026)\textsubscript{o}. The substrate peak cannot be seen in the scan as it lies in lower perpendicular component, but its parallel component is indicated by a black arrow in the figure. The data fit a structural model in which the films are orthorhombically distorted, similar to the bulk material, but keep coherence along the [100] or [010] directions of cubic substrate.
The orthorhombic $a_o$, $b_o$ and $c_o$ lattice parameters can be extracted from the $(116)_o$ and $(206)_o$ reflections shown above. Figure 3.8 shows the evolution of the lattice parameters as a function of thickness for the films grown with $pO_2= 0.9$ mbar and $pO_2= 0.25$ mbar. Both series display similar trend, showing that the oxygen vacancies, which are likely to be present in the films grown at the lowest pressure (giving rise to slightly larger $c_o$ values), do not change the general behaviour. The orthorhombic distortion is the smallest for the thinnest films. With increasing thickness, $c_o$ remains unchanged, while $a_o$ decreases and $b_o$ increases toward their respective bulk values. However, the bulk lattice parameters are not reached in a continuous way, and $a_o$ and $b_o$ saturate for a thickness of around 50 nm. Above about 70nm, a new spot appears in the diffraction maps, as shown in Figure 3.7 (b). This second spot corresponds to a relaxed unit cell with bulk lattice parameters (marked with stars in Figure 3.8). The fact that, in the films grown at 0.9mbar, this spot appears with lattice parameters almost identical to the bulk ones, strongly indicates that these films are stoichiometric, as discussed above. The lattice parameters for the thinnest film grown at 0.25mbar were extracted from grazing incidence diffraction, as shown in figure 3.9 (a). This figure shows that the film is fully coherent with the substrate and it is, thus, tetragonal.

We have shown that the films are orthorhombic (with the exception of the very thin tetragonal ones), but keep coherence with the cubic substrate along the [010]$_c$ or [100]$_c$ directions. Thus, the orthorhombic lattice parameters of the films fulfill $a_o^2 + b_o^2 = (2a_{STO})^2$ for all thicknesses of strained films. From the lattice parameters shown in figure 3.8, the orthorhombic distortion [102], defined by the angle $\gamma$ (see figure 3.9 (b)), can be calculated using the relationship $\cos ((a_o^2-b_o^2)/(a_o^2+b_o^2))$. This is plotted along with the pseudo-cubic lattice parameters, obtained from the values of figure 3.8. As also seen in the reciprocal space maps, the pseudo-cubic lattice parameters for the strained films correspond to that of the SrTiO$_3$ substrate, whereas the orthorhombic distortion increases with increasing thickness, as seen by the decrease in the angle $\gamma$ with increasing thickness. Moreover, in the relaxed part of the 80nm film, both the pseudo-cubic lattice parameters and the angle $\gamma$ are relaxed to their
Figure 3.8: Orthorhombic lattice parameters \((a_o,b_o,c_o/\sqrt{2})\) as a function of thickness for films grown with \(pO_2= 0.9\) mbar (closed symbols) and 0.25 mbar (open symbols). The symbols marked with a star are those of the relaxed part of the layers and correspond to the orthorhombic bulk lattice parameters [39].

From the pseudo cubic lattice parameters of the films, we calculate a Poisson ratio of 0.397 and 0.316 for the films grown at 0.9 mbar and 0.25 mbar, respectively. These values are comparable to those found for other manganites thin films: 0.35 for LSMO/STO [103] and 0.39 for LCMO/STO [104]. The lattice parameters of the films respect the relation \(c_o/\sqrt{2}<a_o<b_o\), for a bulk Jahn-Teller system, suggesting that these distortions are also present in the films. It is worth noting that the orbital ordering takes place at 1500K, above the growth temperature.

In order to better understand the unusual in-plane coherence of the films with the substrate, observed by out-of-plane diffraction measurements and the domain structure of the film, GIXD experiments were performed using synchrotron radiation. Figure 3.10 (a)-(d) shows in-plane RSM’s around selected reciprocal lattice points of the
3.4. Structure and microstructure.

Figure 3.9: (a) Grazing incidence x-ray diffraction for a 2nm TbMnO$_3$ thin film grown at 0.25mbar around the (110)c. The unit area normalized to the reciprocal lattice units of SrTiO$_3$. (b) Schematic drawing of the relationship between the orthorhombic and pseudo-cubic lattice parameters with the substrate lattice. (c) Pseudo-cubic angle ($\gamma$) and pseudo cubic lattice parameter ($a_{pc}$) as a function of thickness for the films grown at 0.9mbar. The bulk values are marked with a star.

SrTiO$_3$ lattice for a 8 nm thin film grown at 0.25mbar.

In all the maps, the central sharp spot corresponds to the position of the SrTiO$_3$ Bragg peak and the other broader visible reflections around it are from the TbMnO$_3$ film. As shown in (a), four broad peaks can be seen around the (110) substrate spot. They correspond to the different equivalent orientations of the orthorhombic unit cell (see sketch in figure 3.12). The presence of these four domains maintains in the film the four-fold symmetry of the cubic substrate. Moreover, as shown in figure 3.10(b) and (c), a peak broadening in the perpendicular direction (along the [010] for the (100) reflection and along [100] for the (010) reflection), the so-called rocking curve, is seen around the central cubic position. This corresponds to the part of the film that is coherent with the substrate (with lattice spacing 3.905 Å). As seen in 3.10 (d), around the
Figure 3.10: RSMs of a 8nm TbMnO$_3$ film grown at 0.25mbar around: (a) the (110)$_c$; (b) the (010)$_c$; (c) the (100)$_c$ and (d) the (220)$_c$ SrTiO$_3$ Bragg reflections. The axes are in reciprocal lattice units of the substrate (1 r.l.u. = 2π/3.905 Å).

(220)$_c$, only two of the four peaks can be observed, those corresponding to the (400)$_o$ reflections. The absence of the (040)$_o$ peaks is due to the relative intensities of those reflections. Indeed, the intensity ratio $I(200)_o/I(020)_o$ is 1.4 in bulk, whereas a ratio of 77 is found for $I(400)_o/I(040)_o$. This makes the intensity of the (040)$_o$ too weak to be detected.

GIXD experiments were also performed for a 55nm film grown at 0.25mbar. Figure 3.11 (a)-(d) shows different in-plane RSMs for this thicker film. From (a), only two film reflections are observed around the substrate (110) peak (which in this case is not visible due to the grazing incidence geometry and the larger thickness of the film). The peaks correspond to the (200)$_o$ (higher H and K values) and the (020)$_o$ (at lower H
3.4. Structure and microstructure.

Figure 3.11: RSMs for a 55nm TbMnO$_3$ film grown at 0.25mbar around (a) the (110)$_c$; (b) the (010)$_c$; (c) the (100)$_c$ and (d) the (220)$_c$ SrTiO$_3$ Bragg reflections. The axes are in reciprocal lattice units of the substrate (1 r.l.u. = 2$\pi$/3.905 Å)

and K values. Analysis of these diffraction maps indicates that, along with the change of $a_o$ and $b_o$, a gradual rotation of the unit cell also occurs at larger thickness, so that the orthorhombic axes ($a_o$ and $b_o$) align parallel to the [110] of SrTiO$_3$ (see figure 3.12). The extremely broad peak, in figures 3.11 (a) and (d), is due to domains existing at all orientations in between those sketched in figure 3.12 (a)-(b) and those in figure 3.12 (c).

The coherence with the substrate in the thinner films is then given by [110]$_o$ parallel to [100]$_c$ (or to [010]$_c$). By further increasing the thickness, the strain energy has increased such that the coherency along the [100]$_c$ cannot be kept and the films relax to their bulk structure, with the orthorhombic in-plane axis parallel to the [110]$_o$ of
Figure 3.12: (a) and (b) Sketch of the four types of orthorhombic domains present in the thinner films and their coherence with the substrate. (c) Sketch of the orthorhombic domains present in the thicker films and their coherence with the substrate. (d) Coexistence of different rotated domains with increasing thickness.

SrTiO$_3$ (see figure 3.12 (c)). The superposition of (a) and (b) reproduces the shoulders seen in the phi scan of figure 3.4, around the peaks separated by 90°. In order to have more insight about the domain structure of the films and in an attempt to elucidate this observation, electron microscopy was utilized.

In figure 3.13, we show TEM plane view images recorded in different samples with thicknesses ranging from 17nm to 140nm, grown at 0.25mbar and with thicknesses of 30nm and 60nm, for films grown at 0.9mbar. In the plane view image of the 17nm TbMnO$_3$ film, two differently oriented types of domains can be seen, along with strain contrast within each orientation. This is consistent with the four types of domains seen via XRD. For the thicker film, two orientations can still be seen. Moreover, depending on the tilt angle (not shown here), strain contrast within a domain can be evidenced, confirming that the films are strained up to 67nm.

A comparative look at the samples grown at 0.25mbar shows that the size of the domains increases with increasing thickness. This means that the number of domain walls per unit area decreases with increasing thickness. The films grown at 0.9 mbar
Figure 3.13: Plane view TEM pictures of a 17nm (a), 34nm (b), 67nm (c) and 140nm (d) TbMnO$_3$ thin film grown at 0.25mbar. (e) and (f) Plane view TEM pictures of a 30nm and a 60nm TbMnO$_3$ thin films grown at 0.9mbar.

Figure 3.14 shows the electron diffraction patterns for a 17nm (a) and a 67nm (b) TbMnO$_3$ film grown at 0.25mbar, obtained in a plane-view geometry. The patterns confirm what has been seen in x-ray diffraction: for the thinner film the diffraction...
peaks are the superposition of four TbMnO$_3$ domains along with the contribution from the substrate; for the 67nm TbMnO$_3$ thin film, the electron diffraction pattern shows a gradual rotations of the domains, as discussed above. This confirms the gradual angular distortion occurring as the thickness is increased, concluded from x-ray diffraction experiments.

Cross-section TEM images for a film having a thickness of 67nm and for a film having a thickness of 140nm, both grown at 0.25mbar, are shown in figure 3.15 (a) and (b). The pictures evidence a dense structure with column-like shape for the 67nm film, whereas clear grain boundaries can be seen for the 140nm film. Moreover, strain contrast can clearly be seen on the 67nm film, at the interface with the substrate, which are absent for the relaxed 140nm film, showing the strained character of the films, despite the evolution of the lattice parameters with thickness.

In order to quantify the evolution of the domain walls with increasing thickness, the number of domains walls within an area of a chosen size were counted$^2$ for the samples grown at 0.25mbar. This quantitative analysis is shown in figure 3.16, where the number of domain walls per unit area is plotted as a function of the inverse of the thickness of the films and indicates a linear trend.

The number of domain walls per unit area for the 140nm thick film, which is relaxed and shows clear grain boundaries rather than a domain morphology, differs

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$^2$The statistical analysis of the TEM images, as well as the TEM images, and the results of figure 3.16 are performed by Sriram Venkatesan (Materials science research group, Zernike Institute for Advanced Materials).
3.5 Conclusions

Figure 3.15: (a) Cross-section TEM images for a TbMnO$_3$ thin film having a thickness of 67nm and grown at 0.25mbar. (b) Cross-section TEM images for a TbMnO$_3$ thin film having a thickness of 67nm and grown at 0.25mbar.

Figure 3.16: Number of domains walls per unit area as a function of the inverse of the thickness.

from the linear trend suggested for the range of investigated thicknesses.

3.5 Conclusions

We have successfully deposited epitaxial orthorhombic TbMnO$_3$ films on (001)-SrTiO$_3$, with thicknesses ranging from 8nm to 80nm. The crystal structure of the thin strained films has been identified as a less distorted orthorhombic unit cell compared to the
bulk one with $a_{film} > a_{bulk}$, $b_{film} < b_{bulk}$ and $c_{film} > c_{bulk}$. We found that the films grow with the c-axis out of plane and they orient themselves in the plane such that the $\langle 110 \rangle_o$ directions align with the $\langle 100 \rangle_c$ directions of the substrate. The orthorhombic lattice parameters $a_o$ and $b_o$ are constrained by epitaxy so that $a_o^2 + b_o^2 = (2a_{STO})^2$. This allows for four equivalent orientations and, therefore, four types of domains are observed, such that the in-plane diffraction patterns display four-fold symmetry. With increasing thickness, the in-plane orthorhombic axes gradually change toward their bulk values, increasing the orthorhombicity of the films, but still keeping the partial coherence and epitaxy relation $a_o^2 + b_o^2 = (2a_{STO})^2$. Due to this, the amount of strain is unchanged up to thicknesses of about 60nm for films grown at 0.9mbar and above 100nm for films grown at 0.25mbar, with the out-of-plane lattice parameter of the TbMnO$_3$ remaining constant. Above this thickness, the lattice relaxes to the bulk one. The possibility of decreasing the orthorhombic distortion via epitaxy, without involving composition changes, or of tuning the orthorhombicity by simply changing the thickness of the film, is of general interest, not only for manganites, but also for vanadates, nickelates and other perovskites, whose magnetic and orbital states are known to strongly depend on the degree of orthorhombic distortion. Finally, the number of domain walls per unit area could be quantified for the films grown at 0.25mbar, by an analysis of the TEM plane view images, and shows an increasing number of domain walls per unit area as the thickness is decreased. This (inverse) linear thickness dependence of the domain wall density is of crucial importance for the results presented later in this thesis.