Paper

Influence of Gas Pressure on Sputtering Deposition of Epitaxial BaTiO₃ Thin Films

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Heteroepitaxial $BaTiO_3$ thin films were prepared by RF magnetron sputtering under various gas pressures ranging from 0.08 to 27 Pa. Influence of the gas pressure was investigated through characterization of X-ray diffraction analyses and ferroelectric properties of the epitaxial $BaTiO_3$ films. When the substrates were placed at a position directly above the sputtering target and the epitaxial films were grown under lower gas pressures, the epitaxial films were crystallographically damaged and no ferroelectricity was observed due to ionic bombardment of particles which are generated at the surface of the target and accelerated by plasma sheath voltage. When the films were grown under higher pressures, the lattice misfit strain was relaxed and ferroelectric properties were degraded, because of a lack of assistance from the irradiated sputtering particles. Sputtering under intermediate gas pressure of 6.3 Pa resulted in optimized crystallinity and ferroelectric properties, in which lattice misfit strain and remanent polarization, $2P_p$, were 5% and 43 μ C/cm², respectively. We concluded that control of energy and/or amount of the irradiated particles at the film surface during heteroepitaxial growth is essential to improve ferroelectric properties of BaTiO₃ thin films.

KEYWORDS: barium titanate, heteroepitaxial growth, high-pressure sputtering, ferroelectricity E-mail address: takaaki.yasumoto@toshiba.co.jp

1.Introduction

It has been reported that lattice misfit strain is accompanied by significant changes in ferroelectric properties of epitaxial $BaTiO_3$ films. $^{1-13)}$ When they are deposited by radio frequency (rf) magnetron sputtering, the epitaxial $BaTiO_3$ films can be grown without relaxation of misfit strain until 200 nm in thickness. In addition, it has been confirmed that the Curie temperature can be artificially increased by more than 100°C , and remnant polarization can be enlarged.

In the sputtering deposition of oxide films, however, negative oxygen ions, which are irradiated from the target surface and accelerated by plasma sheath voltage in the direction opposite from that of positive argon ions, are thought to damage the deposited thin films. $^{14-25)}$ Such damage can be prevented when substrates are placed far from the eroded area of the sputtering target. Nevertheless, from the viewpoint of production, it is preferable to obtain the epitaxial films with high quality at the position directly above the target. The authors have assumed that adopting the method of sputtering at high gas pressure is effective in moderating the high-energy sputtering ions. The crystallinity and ferroelectric properties of the BaTiO₃ thin films were improved by employing a high gas pressure of p = 6.3 Pa during deposition.

The purpose of this study is to investigate the influence of gas pressure over 6.3 Pa until lattice misfit strain relax during sputtering deposition on ferroelectric properties of epitaxial BaTiO₃ films. Substrates were placed at various positions in the sputtering apparatus to acquire information about distribution of the high-energy negative ions emitted from the target and scattered by the ambient gases with particular

pressure.

2. Experimental

Ferroelectric thin-film capacitors which consisted of a SrRuO₃ bottom electrode layer, a BaTiO₃ ferroelectric layer and a SrRuO₃ top electrode layer were prepared on SrTiO₃ (100) single- crystal substrates by radio-frequency magnetron sputtering (model CFS-8EP-55SC, Shibaura Mechatronics Corp.). These layers were sequentially deposited, without breaking the vacuum, to thickness of 30 nm, 50 nm and 30 nm, respectively. The sputtering gas pressure was changed from 13 Pa to 27 Pa. In a range of sputtering gas pressure over 6.3 Pa, the conductive valve was closed and a bypass valve with a lower conductance was used to restrict the pumping rate.

Regarding the sputtering conditions of BaTiO₃ films, the substrate temperature was 650 °C, and the flow ratio of gases was maintained at $Ar:O_2 = 4:1$. The rf power of 90 W was supplied to the target for the lower pressure from 0.08 Pa to 6.3 Pa. The rf power was increased to 200 W or 300 W for the higher pressure of 13 Pa or 27 Pa, respectively, to maintain the same deposition rate as the lower-pressure conditions.

For the deposition of $SrRuO_3$ films, a polycrystalline $SrRuO_3$ target with 4-inch diameter (Kojundo Chemical Laboratory Co., Ltd.) and for the deposition of $BaTiO_3$, a polycrystalline $BaTiO_3$ target with the same diameter were used. The substrate temperature, the gas ratio and the radio frequency power were set to be 650 °C, $Ar:O_2 = 4.1$ and 300 W, respectively. Other aspects of the sputtering procedure, such as substrate positioning in the vacuum chamber have been described in detail in a previous paper.³⁾ As illustrated in Fig. 1, the substrate position

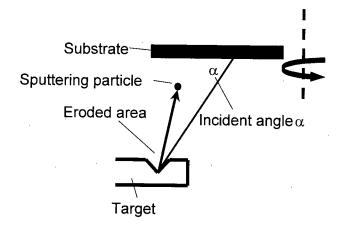


Fig. 1. Schematic diagram of relationship between substrate and eroded area of sputtering target. Location was represented by the incident angle α .

was defined by an incident angle α , with which particles collide at the surface of the substrate from the sputtering target, in the case that they are not scattered by the ambient.

X-ray diffraction analyses were carried out to examine the lattice constant and crystal orientation of the epitaxial films. Two-dimensional reciprocal space maps of the diffraction intensity were measured using a Philips X'pert-MRD system. To measure electrical properties, the top electrode layer was etched off to form thin-film capacitors with an area of 50 μ m x 50 μ m. The bottom SrRuO₃ layer was electrically contacted at regions where BaTiO₃ films were removed selectively by chemical etching. Polarizations vs voltage (P-V) hysteresis loops were monitored with a measuring circuit employing the virtual ground technique. Details of the preparation and measurements have been described in previous papers. $^{1-12}$)

3. EXPERIMENTAL RESULTS

3.1 X-ray Diffraction X-ray diffraction measurements using a θ - 2θ method were carried out for epitaxial BaTiO₃ thin films deposited with various gas pressures from 0.08 Pa to 27 Pa.

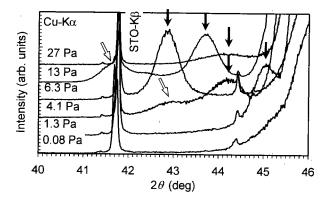


Fig. 2. X-ray diffraction profiles for heteroepitaxial BaTiO₃ films prepared directly above the sputtering target with various sputtering gas pressures.

Figure 2 shows the diffraction profiles around the angle of 2θ from 40° to 45.5° for the specimens placed directly above the target. As indicated by arrows in Fig. 2, diffraction peaks from the BaTiO₃ (002) plane, were clearly observed for the films deposited with a pressure ranging from 1.3 Pa to 13 Pa. The peak was very weak for the BaTiO₃ film deposited with a pressure of 27 Pa. For the films sputtered at 4.1 Pa and 13 Pa, the diffraction peak was split into two peaks, a main-peak and a sub-peak, as indicated by black and white arrows, respectively. It is thought that the BTO film can be divided into two parts, a part with large lattice misfit strain and the other part with a relaxed strain. The diffraction peaks were diversely located ranging from 2θ =42° to 45°, depending on the gas pressure.

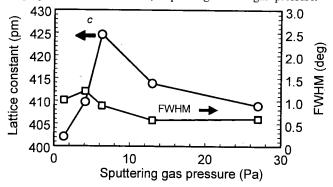


Fig. 3. Lattice constant (c-axis) and full-width at half maximum (FWHM) value of rocking curve of BaTiO₃ films prepared directly above the target (α =86°) as a function of sputtering gas pressure.

The lattice constant c-axis and the full-width at half maximum (FWHM) values of the rocking curves were determined for the BaTiO₃ (002) main peak, and plotted as a function of gas pressures in Fig. 3. The c-axis was maximized at 6.3 Pa and the FWHM values decreased as sputtering pressure increased when the substrates were placed directly above the target.

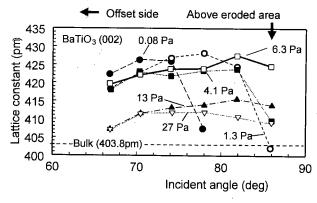


Fig. 4. Incident angle dependence of the lattice constant (c-axis) of heteroepitaxial BaTiO₃ films for each sputtering gas pressure.

Figure 4 shows the lattice constant of the $BaTiO_3$ films deposited with various gas pressures from 0.08 Pa to 27 Pa as a function of substrate position expressed by incident angle α of particles. When the films were deposited with lower pressures

ranging from 0.08 Pa to 4.1 Pa, elongated c-axis was obtained only for the specimens placed at offset positions represented by lower incident angles. The c-axes at the offset positions were more than 4% longer than the inherent c-axis of bulk (403.8 pm). When the pressure was as low as 0.08 Pa, diffraction peak disappeared for the specimen placed directly above the target.

When the sputtering gas pressure was increased to 1.3 Pa, the ${\rm BaTiO_3}$ (002) diffraction peak could be observed over the entire range of incident angles. Although the c-axis exhibited elongated values of 418.3-428.1 pm at the offset positions, it steeply shrank to 402.1 pm at the positions directly above the eroded area.

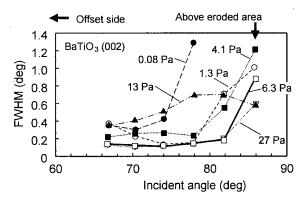


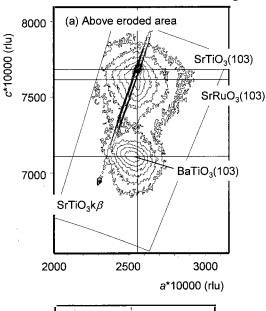
Fig. 5. Incident angle dependence of full-width at half maximum (FWHM) value of rocking curve in heteroepitaxial BaTiO₃ films.

When the sputtering gas pressure was increased to 6.3 Pa, the c-axis was elongated over the entire range of the incident angle. Even when the substrate was placed at the position directly above the target, the c-axis was 424.6 pm, which is 5.3% longer than that of bulk. For the gas pressures of 13 Pa or higher, the c-axis of main peak was relaxed over the entire range of the incident angle. When the substrate was placed at the position directly above the target, the c-axis was 408.9 pm at 27 Pa, which is only 1.4% longer than that of bulk.

The c-axes of the epitaxial BaTiO3 films were maximized at different incident angles depending on the sputtering gas pressure. For higher gas pressures, the maximum value of the c-axis appeared at positions closer to the target, where it is indicated by higher incident angles. When the sputtering gas pressure was 6.3 Pa, the best uniformity was obtained, probably due to moderation of the high-energy particles by scattering. The longest c-axis was obtained at 6.3 Pa for the film positioned close to the eroded area. For high gas pressure of 27 Pa, however, the maximum value of the c-axis appeared at lower incident angles again, as observed at the lower sputtering pressure. This result suggests that negative particles, such as oxygen ions, with moderate-energy have a role in preventing relaxation of lattice misfit strain. At a higher gas pressure, the particles are scattered by the ambient molecules, and their energies were declined.

In the case that two diffraction peaks were observed, as for the pressure of 4.1 Pa or 13 Pa, the rocking curve was measured for the main-peak. Figure 5 shows the FWHM values of the rocking curves plotted as a function of the incident angle, α . For all gas pressures, the FWHM values of the rocking curves were smaller for the specimens placed at the offset area, which was distant from the target.

The incident angle where the FWHM value suddenly increased approximately agreed with the angle where elongation of the lattice constant c-axis abruptly relaxed for specimens prepared below 4.1 Pa. The value of FWHM directly above the eroded area tended to be smaller for higher sputtering



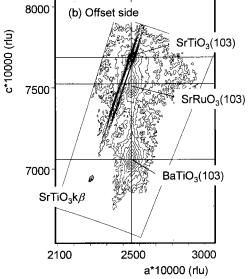


Fig. 6. Reciprocal space mappings of X-ray intensity around the (103) reflection for heteroepitaxial BaTiO₃ films prepared (a) directly above the target (α =86°) and (b) at an offset position (α =70°). Four contour line indicate 10 times in X-ray intensity.

pressure. When the sputtering gas pressure was increased to 27 Pa, the FWHM values were as low as 0.2° over a wide range of incident angles except for directly above the eroded area, where the FWHM increased to 0.57°. The crystals seem to be

damaged by the incident particles, even when the pressure was as high as 27 Pa.

Reciprocal space mapping of X-ray intensity around a (103) plane was measured for the BaTiO₃ films prepared both directly above the target (α =86°) and at an offset position (70°), as shown in Figs. 6 (a) and 6 (b), respectively. The BaTiO₃ and SrRuO₃ peaks had almost the same α -axis with the SrTiO₃ substrate. This indicates that the lattice misfit strain of the BaTiO₃ films did not relax significantly during the epitaxial growth. Clear difference was observed, however, in strength and broadness of the diffraction peaks for both BaTiO₃ and SrRuO₃ films between the substrate positions. Uniformity of the crystal orientations was degraded for the films prepared directly above the target.

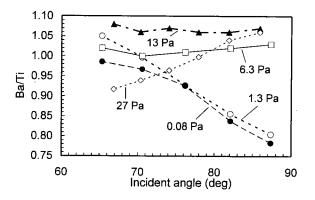


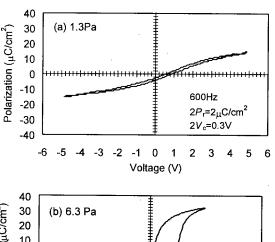
Fig. 7. Incident angle dependence of heteroepitaxial BaTiO₃ film composition ratio for Ba/Ti for each sputtering gas pressure.

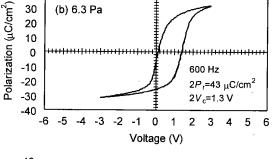
3.2 Compositional analysis The sputtering gas pressure also influenced the composition of the BaTiO₃ thin films. The atomic ratio Ba/Ti was analyzed by inductively coupled plasma atomic emission spectrometry (ICP-AES) and plotted as a function of the incident angle α for the BaTiO₃ films deposited under various sputtering gas pressures, as shown in Fig. 7.

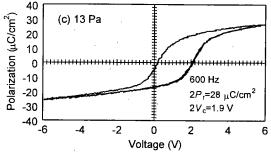
In the case that the pressure was as low as 0.08 Pa or 1.3 Pa, the composition was Ba-poor (Ba/Ti < 0.9) at the positions directly above the target. It has been reported that composition deviates from stoichiometry as a result of the spatial distribution of sputtered particles. $^{\rm 18}$ When the pressure was increased to 6.3 Pa, however, the composition approached stoichiometry (0.9 < Ba/Ti < 1.05) over a wide range of the substrate positions. The uniform composition might be a result of scattering of sputtered particles in ambient gases.

When the sputtering gas pressure was increased to 13 Pa or 27 Pa, the composition deviated from stoichiometry to Ba-rich (Ba/Ti > 1.05) at the positions directly above the target. This tendency is opposite to that observed at low gas pressures.

3.3 Ferroelectric properties Polarization vs. voltage (P-V) hysteresis loops were measured for the epitaxial BaTiO₃ thin films, which were placed at different positions in the vacuum chamber, and deposited with various gas pressures.







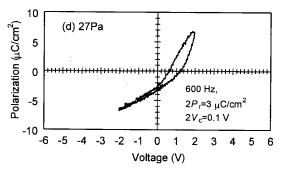


Fig. 8. Polarization vs voltage (P-V) hysteresis loops observed for heteroepitaxial BaTiO₃ films prepared at sputtering gas pressures of (a) 1.3 Pa, (b) 6.3 Pa, (c) 13 Pa, and (d) 27 Pa.

Hysteresis loops, which were measured for specimens prepared directly above the target with various gas pressure (1.3 Pa to 27 Pa), are shown in Figs. 8(a) to (d).

When the epitaxial films were grown with a gas pressure of 1.3 Pa, ferroelectricity was very weak. When the epitaxial films were grown at 6.3 Pa, clear hysteresis loop was observed. When the epitaxial films were grown with a gas pressure of 13 Pa, hysteresis loop could be observed, whereas its shape inclines. With a high gas pressure of 27 Pa, the polarization could not be

measured until sufficient saturation because of leakage current.

Since the centers of the hysteresis loops were deviated from the origin of the voltage axis (V=0) for all specimens, the ferroelectricity was evaluated at the maximum of the remnant polarization, $2P_{rmax}$. The remanent polarization was plotted as a function of the incident angle for the films with various sputtering gas pressures, as shown in Fig. 9.

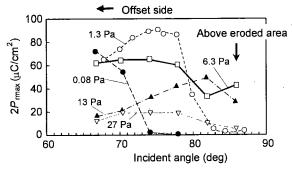


Fig. 9. Incident angle dependence of remnant polarization, $2P_{rmax}$, for epitaxial BaTiO₃ films sputtered under various gas pressures.

When the films were prepared at a low gas pressure of 0.08 Pa, the remanent polarization $(2P_{\text{rmax}})$ was maximized at an offset position far from the eroded area, and rapidly decreased at a higher incident angle (α =70°). When the gas pressure was 1.3 Pa, the maximum value of $2P_{\text{rmax}}$ was as large as 91 $\mu\text{C/cm}^2$. However, the remanent polarization also sharply declined at a higher incident angle (α =80°). When the pressure was increased to 6.3 Pa, no degradation of the remnant polarization was observed even at the highest incident angle (α =86°). When the pressure was 13 Pa, the remnant polarization decreased in both above eroded area and offset positions. This result corresponds to the shortening of the lattice constant of c-axis in each substrate position as shown in Fig. 4 and the decreasing of the FWHM as shown in Fig. 5. When the pressure was increased to 27 Pa, degradation of the remnant polarization propagated through all positions.

4. Discussions

The ferroelectric properties of the BaTiO₃ thin films were optimized by employing a pressure of 6.3 Pa, and deteriorated at gas pressures over 6.3 Pa for the most substrate positions. In the literature concerning sputtering deposition of oxide films such as ZnO film or YBaCuO film, improvement of crystallinity has been reported when sputtering gas pressure was sufficiently high¹⁴⁻²⁵⁾. Energetic particles such as oxygen negative ions are considered to lose their kinetic energy by scattering before they reach the surface of the substrates with the ambient gas molecules when pressure is high.

Because the purpose of this study is to obtain epitaxial BaTiO₃ films with better ferroelectricity and larger remnant polarization, the magnitude of the lattice misfit strain was an essential factor. From this viewpoint, favorable ferroelectric properties were only realized for epitaxial BaTiO₃ films when they were deposited under appropriate conditions.

In the epitaxial BaTiO3 films, existence of the incidence

particle of moderate energy promoted the growth of the lattice constant of c-axis, which also improved ferroelectric properties. If incidence particles had less energy, the lattice constant of c-axis would easily relax, and ferroelectricity would also be weakened.

When the sputtering of the epitaxial BaTiO₃ film was carried out at the high gas pressure, the crystallinity expressed by the FWHM of the rocking curves was not improved significantly above the eroded area. A possible reason is that the crystallinity was degraded by introduction of misfit dislocations, because the negative ions lost their energy by scattering in ambient gas molecules at high pressures. At the pressure of 27 Pa, however, the FWHM was large only at the position directly above the target, whereas the lattice constant of c-axis did not vary significantly compared to the other positions. suggests that another factor is working for the deterioration of For example, a quite small number of crystallinity. high-energy particles may collide into the film surfaces without scattering even at 27 Pa, and cause destruction of the crystals placed directly above the target. Although continuous irradiation of moderate-energy particles is required to prevent relaxation of lattice misfit strain throughout epitaxial growth of the BaTiO3 films, bombardment by a small number of extraordinarily high-energy particles may cause a serious damage to their crystallinity.

5. Conclusions

In the sputtering deposition of epitaxial BaTiO₃ thin films, it was confirmed that ambient gas pressure has a significant influence on the ferroelectricity of BaTiO₃ films. When the epitaxial films were grown under lower gas pressures, the films exposed to ionic bombardment were so seriously damaged that no ferroelectricity was observed. When the sputtering gas pressure was increased to 6.3 Pa, the ferroelectric properties were improved at a wide range of substrate positions. When the pressure was increased to over 6.3 Pa, remnant polarization of *P-V* hysteresis loops was degraded. These results are closely related to the energy of particles, such as the oxygen negative ion, which collide into the substrate surface,

In conclusion, in order to optimize the ferroelectric properties in epitaxial BaTiO₃ thin films, control of both the kinetic energy and the amount of the negative oxygen ions irradiated into the surface of the films, is considered to be essential.

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