Epitaxial growth of neodymia stabilized zirconia on Si(001) substrate using dynamic aurora PLD

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Based on previously reported studies, yttria stabilized zirconia \([Y_{0.15}Zr_{0.85}O_{1.93}} (YSZ)\] has been used as an epitaxial buffer layer on Si(001) substrates. However, a considerable lattice mismatch exists between the YSZ and Si (−5.4%). This work elucidates the reported relationship between the lattice parameter and composition of neodymia stabilized zirconia \([\text{Nd}_{X}Zr_{1−X}O_{2−Y/2}} (NdSZ)\]. According to the relation, the lattice parameter of NdSZ is the same as that of Si at \(x = 0.75\). Therefore, if the epitaxial growth of NdSZ thin film with \(x = 0.75\) is realized on a Si(001) substrate, then zero lattice mismatch can be expected. The deposition of NdSZ thin film on Si(001) substrate was done by dynamic aurora pulsed laser deposition (PLD). Results show that cube-on-cube epitaxial growth \((\text{NdSZ(001)}[\text{100}] \// \text{Si(001)}[\text{100}]\) is realized between \(x = 0.16\) and \(x = 0.52\). The best composition of epitaxial NdSZ thin film is regarded as \(x = 0.47\) from the point of crystallinity, orientation and lattice mismatch (−1.1%). When composition \(x\) is larger than \(x = 0.57\), the NdSZ thin film orientation becomes (111). Results also show that the lattice parameter of the NdSZ thin film is larger than that of NdSZ bulk. For \(x = 0.47\) thin film, the coexistence of the epitaxial (221) domains is detected in addition to the (001) cube-on-cube domain. The (221) domain is formed due to lattice matching of the oxygen sub-lattice, which has an isosceles triangle shape. We produced cube-on-cube epitaxial NdSZ thin film on Si(001) substrate with a very small lattice mismatch. However, such a small lattice mismatch brings about additional (221) domains that have been reported in the epitaxial growth of CoSi2 thin film on Si(001) substrate. The lattice mismatch between CoSi2 and Si is −1.2%, which is approximately equal to that between NdSZ \((x = 0.47)\) and Si.

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1. Introduction

Epitaxial integration of oxide on Si(001) has been investigated to realize future oxide electronics such as ferroelectric random access memories,1,2 superconductors,3 magnetic devices,4 new-generation radio frequency filter application5, and multiferroic applications.6,7 Reportedly, only a few oxides enable direct epitaxial growth on Si(001), such as yttria stabilized zirconia \([Y_{0.15}Zr_{0.85}O_{1.925}} (YSZ)]\)5, BaO,7 SrO,10 MgO,11 and SrTiO3.12,13 In the case of SrTiO3, earlier reports have described that the deposition of thin SrO layers before the deposition of SrTiO3 thin film is extremely important for the realization of epitaxial growth of SrTiO3 on Si(001).14 Therefore, epitaxial SrTiO3 on Si(001) should be described as SrTiO3/SrO/Si(001). Hence one might reasonably infer that the oxides which enable direct epitaxial growth on Si(001) are limited to YSZ, BaO, SrO, and MgO among the oxides described above. Actually, among these oxides, the chemical stability of BaO, SrO, and MgO is extremely low. These react rapidly to form carbonates or hydroxides in the air. Therefore, in the case of these oxides, subsequent deposition of chemically stable oxide overlayer such as SrTiO3 is needed without breaking vacuum. However, YSZ is chemically very stable. Therefore, to realize subsequent epitaxial growth using another deposition method such as chemical solution deposition, handling epitaxial YSZ thin films on Si(001) can be done easily. Therefore, YSZ has been used extensively as a promising epitaxial buffer layer on Si(001). However, YSZ has an important shortcoming that the lattice parameter \((a = 0.5139 \text{ nm})\) is

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smaller than that of Si (a = 0.543088 nm). The lattice mismatch between YSZ and Si is −5.4% as calculated using the following formula.15)

\[
\frac{a_{\text{thin film}} - a_{\text{substrate}}}{a_{\text{substrate}}} \times 100\% \quad (1)
\]

This value of lattice mismatch is considerably large from the perspective of the epitaxy. In addition, when preparing epitaxial oxide electrode thin films such as (La, Sr)CoO₃ (LSCO) having (001) orientation on YSZ/Si(001), this epitaxial growth is known not to be realized because of a large lattice mismatch. The lattice parameter of LSCO is a = 0.3805 nm. When the lattice of perovskite LSCO has 45° rotation along (001), the d-spacing after 45° rotation is 0.3805*√2 = 0.5381 nm. In this case, the lattice mismatch between LSCO and YSZ is +4.7%. Because of this great degree of lattice mismatch, it is impossible to produce an epitaxial LSCO thin film on YSZ/Si(001) having (001) orientation. To overcome this shortcoming, a double buffer layer of CeO₂/YSZ/Si(001) has been used widely for epitaxial growth of perovskite oxides such as LSCO.16,17) Lₐ₀₉₅(Sr, Ca)₂₀₃₅MnO₄,18) LaNiO₃,7,19,20) and SrRuO₃21) because the lattice mismatch between CeO₂ and these perovskite-type compounds is very small. For LSCO, the lattice mismatch between CeO₂ and LSCO is only 1.0602 nm. Because of this great degree of lattice mismatch, it is impossible to produce an epitaxial LSCO thin film on YSZ/Si(001) having (001) orientation. To overcome this shortcoming, a double buffer layer of CeO₂/YSZ/Si(001) has been used widely for epitaxial growth of perovskite oxides such as LSCO.16,17) Lₐ₀₉₅(Sr, Ca)₂₀₃₅MnO₄,18) LaNiO₃,7,19,20) and SrRuO₃21) because the lattice mismatch between CeO₂ and these perovskite-type compounds is very small. For LSCO, the lattice mismatch between CeO₂ and LSCO is only −0.6%. The CeO₂/YSZ/Si(001) double buffer layer has also been used for heteroepitaxial growth of oxide superconductor (YBa₂Cu₃O₇)₃,22,23) and ferrite [(Ni, Zn)Fe₂O₄].23)

Although the lattice mismatch between CeO₂ and Si is extremely small (−0.4%), it is expected that CeO₂ thin film can be grown epitaxially on Si(001). However, earlier reports have described that CeO₂ thin film deposited on Si(001) substrate is not (001) but (111) orientation.24) Therefore, in the CeO₂/YSZ/Si(001) structure, the YSZ layer has the role of direct epitaxial growth on Si(001); CeO₂ has the role of reducing lattice mismatch and realizing epitaxial growth of several oxides on it. However, the deposition of two layers on Si(001) is rather time-consuming and costly, especially on an industrial scale. Therefore, the objective of this work is to develop a novel single buffer layer which enables epitaxial growth on Si(001) having a small lattice mismatch. In this work the possibility of neodymia stabilized zirconia [NdₓZr₁₋ₓO₂₋ₓ/2 (NdSZ)] on Si(001) is investigated.

2. Concept of this work

Figure 1(a) shows the relation between the lattice parameter of YₓZr₁₋ₓO₂₋ₓ/2 and Y content x (=Y/(Y + Zr)). In this figure, black closed circles are cited from Popov et al. for a bulk ZrO₂–Y₂O₃ system.25) A blue closed circle is half of the lattice parameter of pure Y₂O₃ [c-type rare earth structure (a = 1.0602 nm)].26) Because the c-type rare earth structure is an ordered derivative of the defect fluorite structure, it is reasonable that the lattice parameter varies linearly with composition between x = 0.15 and x = 1. Although the crystal system of pure ZrO₂ is monoclinic, it would be possible to estimate the lattice parameter of pure ZrO₂ in cubic form by the extrapolation of the relationship between the lattice parameter and composition. This extrapolation is shown by the dotted line in Fig. 1(a). Results demonstrate that the estimated lattice parameter of pure ZrO₂ in cubic form is a = 0.5111 nm, as shown in a red closed circle. In this figure, the lattice parameter of Si and CeO₂ is also shown as two lines. This figure shows that the lattice parameter of YₓZr₁₋ₓO₂₋ₓ/2 is smaller than that of Si for all compositions x.

Figure 1(b) presents the relation between the lattice parameter of NdₓZr₁₋ₓO₂₋ₓ/2 and Nd content x (=Nd/(Nd + Zr)) mol%. In this figure, black closed circles are cited from Lakiza et al. for a bulk ZrO₂–NdO₃ system.27) The red open circle is the half of lattice parameter of pure Nd₂O₃ [c-type rare earth structure (a = 1.1076 nm)].28) In this figure, the lattice parameter of pure ZrO₂ in cubic form estimated from Fig. 1(a) is shown as a red closed circle. When connecting the red closed circle (x = 0) and the red open circle (x = 1) by a line, results show that the relation between the lattice parameter of NdSZ agrees well with this line between x = 0.17 and x = 0.69. This figure reveals that the lattice parameter of NdSZ will be the same as that of Si for x = 0.75. If NdSZ thin film having this composition can be grown epitaxially on Si(001), then the lattice mismatch between NdSZ and Si is expected to be zero. In this work, we examine the possibility of realizing the epitaxial growth of NdSZ thin film on Si(001) having a very small lattice mismatch.

3. Experimental

NdSZ thin films were deposited on Si(001) substrates with native oxide using Dynamic Aurora pulsed laser
deposition (PLD) with a Nd: YAG laser (λ = 266 nm). The sintered ceramic target, synthesized using conventional solid-state reaction, was irradiated by a focused pulsed laser. For the Dynamic Aurora PLD, an electromagnet was installed in the vacuum chamber between a target and a substrate. Therefore, a magnetic field can be applied parallel to the direction from the target to the substrate during thin film deposition. Our earlier work revealed that the application of a magnetic field during deposition brings about a convergence of the PLD plume (plasma) and thereby increases the deposition rate.28),29) For this study, thin film deposition was done under a 200 G magnetic field. Detailed deposition conditions are presented in Table 1. The NdSZ film thickness was 50 nm. The thin film crystal structure was examined using an X-ray diffractometer (XRD: D8 Advance; Bruker AXS GmbH, Germany). The crystal structure of the thin film, including reciprocal space mapping (RSM) measurement and pole figure measurement, was examined using a high-resolution XRD (ATX-G; Rigaku Corp., Japan). For compositional analysis, as a preliminary study, we analyzed the composition of YSZ thin film using X-ray fluorescence (XRF). The analyzed composition of YSZ thin film was Y0.11Zr0.88O1.93, which is very close to the prescribed composition (Y0.11Zr0.85O1.93) within the error of the analysis. For the XRF measurement, the calibration curve is extremely important for the analysis of the accuracy of composition. As the slope of the calibration curve is sensitive to the density of standard samples, metals or single crystals having 100% theoretical density should be used to prepare the calibration curve. For YSZ, a single crystal can be used as the standard sample. However, it is impossible to obtain NdSZ single crystal. In addition, Nd metal is chemically too active to handle in the air. Hence obtaining a reliable calibration curve to analyze the composition of NdSZ thin film is impossible. For this reason, we used the prescribed composition as the thin film composition.

### Table 1. Deposition conditions of NdSZ thin films

| Laser            | Nd:YAG laser (λ = 266 nm) |
|------------------|---------------------------|
| Repetition rate/Hz| 10                        |
| Target           | Nd2Zr1−xO2−x/2 (NdSZ)    |
| Prescribed composition | x = 0.16–0.90 |
| Substrate        | Si(001)                   |
| Fluence/J/cm²    | 2.0                       |
| Deposition time/min| 30                        |
| Magnetic field/G  | 200                       |
| Substrate temperature/°C | 800                |
| Oxygen pressure during deposition/Pa | 7.33 × 10⁻² |

4. Results and discussion

**Figure 2** presents XRD (ω/2θ) patterns of NdSZ thin film with (a) x = 0.35, (b) x = 0.39, (c) x = 0.42, (d) x = 0.47, (e) x = 0.52, (f) x = 0.57, and (g) x = 0.90. The NdSZ(002) for x = 0.57 and 0.90. In this figure, thin films having (001) and (111) orientations are shown as surrounded by dotted ellipses. The lattice parameters of NdSZ thin films with x = 0.47 and 0.52 are, respectively, a = 0.537 and 0.538 nm. These values are very close to the lattice parameter of Si. The lattice mismatches between Si and NdSZ with x = 0.47 and 0.52 are, respectively, −1.1 and −0.9 %. The lattice...
parameters of NdSZ thin films with $x = 0.57$ and 0.90 are, respectively, $a = 0.545$ and 0.547 nm. These values are slightly larger than that of Si. The lattice mismatches between Si and NdSZ with $x = 0.57$ and 0.90 are, respectively, $+0.4$ and $+0.7\%$. Formation of (111) orientation in NdSZ can be explained in two possible ways. One possibility is the similarity with CeO$_2$ thin film on Si(001). As described above, earlier reports show that CeO$_2$ thin film deposited on Si(001) has (111) orientation. The lattice parameter of CeO$_2$ ($a = 0.5409$ nm) is very close to that of Si. In the same way, the lattice parameter of NdSZ with $x = 0.57$ and 0.90 is also very close to that of Si. Therefore, there is a possibility that the (111) orientation would be brought about by the small lattice mismatch for a fluorite structure. Another possibility is related to the free energy of the formation of SiO$_2$. For the epitaxial growth mechanism of YSZ on Si(001) with SiO$_2$, the following equation has been considered.

$$\text{Zr} + \text{SiO}_2 \rightarrow \text{ZrO}_2 + \text{Si}$$

The metallic Zr or reduced Zr ion such as Zr$^{4+}$ is formed by the laser irradiation of PLD. When the $x$ value in NdSZ increases to a certain extent, the concentration of Zr decreases. Then the driving force to bring about Eq. (2) will be lowered, thereby bringing about the formation of SiO$_2$ on the Si(001) surface. In this case, the epitaxial growth of NdSZ is hindered and the energetically most stable (111) plane will be formed.

As depicted in Fig. 3, a NdSZ thin film with $x = 0.52$ has the smallest lattice mismatch with (001) orientation. However, as presented in Fig. 2, the peak intensity of the NdSZ thin film with $x = 0.52$ is much lower than that of the film with $x = 0.47$. Therefore, we consider that the best composition for NdSZ thin film is $x = 0.47$. Figure 3 also shows that the lattice parameter of the NdSZ thin film is larger than that of a reported NdSZ powder having the same composition. The reason is not clear, but two possibilities can be considered. One possibility is that the NdSZ thin films are metastable, having lattice defects such as excess oxygen at the interstitial site. Another possibility is the deviation of the composition of the NdSZ thin film from the prescribed composition described above.

Figure 4 presents reciprocal space maps around NdSZ (113) with (a) $x = 0.16$, (b) $x = 0.37$, (c) $x = 0.42$, and (d) $x = 0.47$. This figure shows that the distance of the peak top between NdSZ(113) and Si(113) decreases with composition $x$. In addition, the $Q_y$ value of NdSZ differs from that of Si, which demonstrates that the NdSZ thin film is relaxed irrespective of the composition. Figure 5 shows the variation of both out-of-plane and in-plane lattice parameters with composition. The out-of-plane and in-plane lattice parameters were determined based on the reciprocal space maps portrayed in Fig. 4. In this figure, the out-of-plane lattice parameter measured from $\omega/2\theta$ scan depicted in Fig. 3 is also shown. The in-plane lattice parameter is almost identical to the out-of-plane lattice parameter. Both lattice parameters are the same as the lattice parameter determined from $\omega/2\theta$ scan, which means that the NdSZ thin film is cubic between $x = 0.16$ and 0.47.

Figure 6(a) depicts (100) pole figure of NdSZ thin film with $x = 0.47$. Figure 6(b) shows a simulation of (100) pole figure of NdSZ thin film having (001) orientation. Comparison of Figs. 6(a) and 6(b) shows that all poles depicted in Fig. 6(b) are apparent in Fig. 6(a). However, in Fig. 6(a), additional weak poles are also detected. These additional poles indicate that the domains having another orientation are also epitaxially grown. Figure 6(c) presents a simulation of (100) pole figure of NdSZ thin film having...
(221) orientation. In Fig. 6(c), simulated patterns of four kinds are depicted, one for every 90° rotation. Figure 6(d) is drawn by superimposing the four kinds of pole figures depicted in Fig. 6(c). By comparing Fig. 6(a) with Fig. 6(d), the additional weak poles in Fig. 6(a) exactly agree with the poles in Fig. 6(d). Therefore, the NdSZ thin film with \( x = 0.47 \) comprises (001) and (221) epitaxial domains. It is important to know that (221) domains cannot be observed by XRD because the peak position in 2\( \theta \) of (221) coincides with that of (003). The sum of the squares of each Miller index of (221) is \( 2^2 + 2^2 + 1^2 = 9 \); that of (003) is \( 0^2 + 0^2 + 3^2 = 9 \).

To explain why (221) domains coexist with (001) domains, the crystal structure of NdSZ is shown in Fig. 7(a). In this figure, \( a_{FL} \) represents the lattice parameter of the fluorite structure. In the figure, the (221) plane is also shown. Figure 7(b) shows an atomic arrangement of the (221) plane of NdSZ. As shown in this figure, the (221) plane comprises the oxygen sub-lattice in the fluorite structure. The (221) plane shape portrayed in Fig. 7(b) is an isosceles triangle. The respective lengths of the two equal sides and the base are \((\sqrt{2}/2)a_{FL}\) and \((\sqrt{2}/2)a_{FL}\). Figure 7(c) portrays the atomic arrangement of (001) plane of Si. In this figure, \( a_{Si} \) represents the lattice parameter of Si. Figure 7(d) presents a schematic image of (001) epitaxial growth (cube-on-cube) between NdSZ and Si. As the figure shows, the (001) domain grows such that the square consisting of Zr and Nd meets with that of Si. Figure 7(e) presents a schematic image of the (221) epitaxial growth between NdSZ and Si. In this figure, the length of the diagonal line of Si(001) \((\sqrt{2}a_{Si})\) agrees with twice the base of the isosceles triangle \(2 \times (\sqrt{2}/2)a_{FL} = \sqrt{2}a_{FL}\). This figure shows four isosceles triangles with every 90° rotation. The arrangement of these four isosceles triangles agrees with the four kinds of (100) pole figures having (221) orientations. The coexistence of (221) domains has never been reported for an epitaxial YSZ/Si(001) thin film. Therefore, the epitaxial relation portrayed in Fig. 7(e) never occurs in the case of YSZ/Si.
with $x = 0.47$. This fact suggests that (001) epitaxial growth portrayed in Fig. 7(d) can be realized in the case of a large lattice mismatch. However, (221) epitaxial growth as portrayed in Fig. 7(e) occurs only in the case of a small lattice mismatch. In other words, (221) epitaxial growth as portrayed in Fig. 7(d) can be realized in the case of a large lattice mismatch with Si(001). This fact suggests that (001) epitaxial growth is extremely slight ($-1.1\%$). Pole figure measurement reveals that the NdSZ thin film is grown epitaxially on Si(001). However, it was also found that the epitaxial growth of the (221) domain also occurs. Consequently, in this work, we achieved the epitaxial growth of NdSZ thin film having a very small lattice mismatch on Si(001). Moreover, results show that small lattice mismatch brings about an epitaxial (221) domain in addition to the cube-on-cube (001) domain because lattice matching in the oxygen sub-lattice, with an isosceles triangle shape, is achieved in addition to lattice matching in the metal sub-lattice, with a square shape.

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| Table 2. Lattice parameter, lattice mismatch with Si, and the coexistence of (221) domain of YSZ, CoSi2, and NdSZ thin film with $x = 0.47$. |
|------------------------|----------------|----------------|----------------|
| Substance | Lattice parameter (nm) | Lattice mismatch with Si (nm) | (221) domain |
|-----------|----------------|----------------|----------------|
| YSZ | 0.5139 | -5.4 | No coexistence |
| CoSi2 | 0.5365 | -1.2 | Coexistence |
| NdSZ ($x = 0.47$) | 0.537 | -1.1 | Coexistence |
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