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Influence of Pb and La Contents on the Lattice Configuration of La-Substituted Pb(Zr,Ti)O_3 Films Fabricated by CSD Method

Hiromi Shima, Ken Nishida, Hiroshi Funakubo, Takashi Iijima, Takashi Katoda, Hiroshi Naganuma, and Soichiro Okamura

***Abstract*—The influence of Pb and La contents on the lattice configuration in La-substituted Pb($Zr_{0.65}$,$Ti_{0.35}$)O_3 (La-PZT) films was systematically investigated. La-PZT films with various La and Pb contents were fabricated on Pt/Ti/SiO_2/Si substrates by chemical solution deposition (CSD). In the La-PZT films with a Pb content ratio of 125% relative to a stoichiometric value, La ions were substituted for not only A-site ions but also B-site ions at La contents greater than 3 mol%. La substitution for B-site seems to cause larger reduction of the unit cell size. In addition, we found that in the La-PZT films with a La content of 3 mol%, the Pb content of 116 mol% (120% relative to a stoichiometric value) was optimum from the viewpoint of site occupancy. This indicates that excess Pb prevented the A-site substitution of La ions.**

I. INTRODUCTION

FERROELECTRIC (Pb,La)(Zr,Ti)O_3 (PLZT) films have attracted much attention for the applications to novel integrated-type optical devices such as waveguides [1], [2], modulators [3], [4], and tunable photonic crystals [5]. Therefore, studies to improve electro-optic properties of the PLZT films continue [6]–[11]. In the case of PLZT bulk ceramics with a Zr:Ti ratio of 65:35, La content is frequently adjusted to be 8 to 9 mol% for optical applications because of high transparency and large electro-optic coefficients [12]. On the other hand, in the case of PLZT films fabricated by chemical solution deposition (CSD) using precursor solutions with some excess Pb elements, we have reported that PLZT films with an La content of 0 mol% showed some transparency and the largest electro-optic effect [9]. However, the reason why the CSD-derived PLZT films showed such different tendencies from bulk ceramics is not yet clear. It is well known that, in the case of PLZT bulk ceramics, La ions are substituted for an A-site ion (Pb^{2+}: 0.119 nm) not a B-site ion (Zr^{4+}: 0.072 nm, Ti^{4+}: 0.0605 nm) because of the ionic radius [La^{3+}: 0.103 nm(6-CN), 0.136 nm(12-CN)]. However, there is a possibility that La ions are substituted for B-site ions in the CSD-derived PLZT films, and the La-substitution state may have some influence on the structure and the properties of PLZT films. Although there are many previous studies of the effect of Pb and La contents on PLZT or PZT films [13]–[16], no one has reported on the substituted sites and the variation in a *d*-space of PLZT films with various compositions. Therefore, in this paper, we systematically investigate the influence of Pb and La contents on the lattice configuration of La-substituted Pb($Zr_{0.65}$,$Ti_{0.35}$)O_3 (La-PZT) films fabricated by CSD.

II. EXPERIMENTAL

La-PZT films were grown on (111)Pt/Ti/SiO_2/(100) Si substrates by CSD. The chemical solutions provided by Kojundo Chemical Laboratory (Saitama, Japan) were used as precursor solutions. We used commercially available precursor solutions. In this study, we prepared La-PZT films with different compositions: La contents ranging from 0 to 15 mol% and excess Pb content ratios relative to stoichiometric values ranging from 10 to 30%. The 10 kinds of La-PZT precursor solutions used in this study are summarized in Table I. In our process, the Pb content ratio of 125% relative to a stoichiometric value was optimum for the La-PZT films with well-filled structure [10]. The La-PZT precursor solutions were spin-coated at 3000 rpm for 50 s. The spin-coated films were dried at 100 to 120°C for 5 min and calcined at 350°C for 5 min. After spin-coating and calcination were repeated 4 times, the films were sintered at 700°C for 10 min in a mixture gas of O_2 and Ar at an Ar: O_2 flow ratio of 95: 5 mL/min by rapid thermal annealing (RTA). Final film thickness varied from 310 to 350 nm depending on La and Pb contents. Precursor solutions with higher La and Pb contents caused thicker films because of higher viscosity.

The crystal structure and orientation of the films were determined by X-ray diffraction analysis (XRD) using PANalytical X'Pert MRD (Almelo, The Netherlands) with CuKα radiation. For the evaluation of electrical properties, top electrodes (0.1–0.4 mm in diameter) made of Pt

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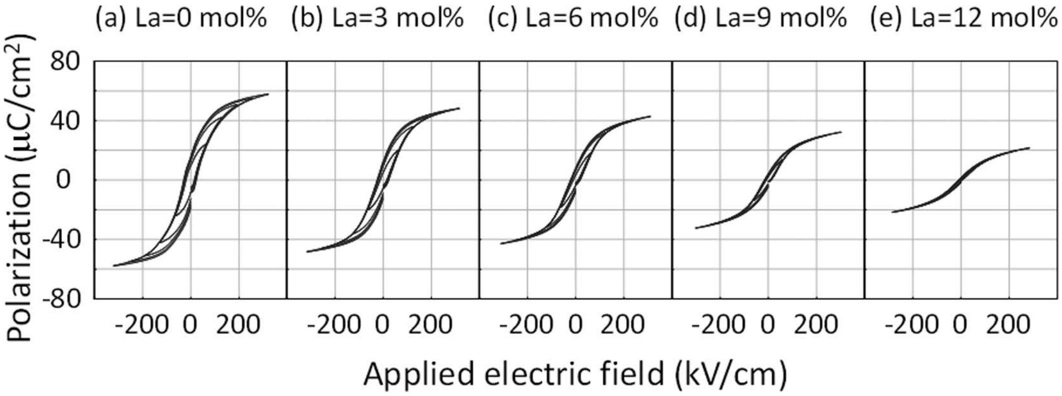

Fig. 1. *D-E* hysteresis loops of La-PZT films with La contents of (a) 0 mol%, (b) 3 mol%, (c) 6 mol%, (d) 9 mol%, and (e) 12 mol%, respectively.

TABLE I. COMPOSITIONS OF TEN KINDS OF LA-PZT PRECURSOR SOLUTIONS USED IN THIS STUDY.

No.	La content mol%	Pb content mol%	Pb content Excess Pb content ratio (%)
1	0	125	25
2	3	121	25
3	6	118	25
4	9	114	25
5	12	110	25
6	15	106	25
7	3	107	10
8	3	112	15
9	3	116	20
10	3	126	30

were deposited on the film surface by rf magnetron sputtering. The *D-E* hysteresis loop of the films was measured using a ferroelectric tester (aixACCT TF-2000) with a single triangular pulse of 100 Hz. The substitution site of La ions was identified by Raman spectroscopy. Raman spectra were measured using a laser Raman spectroscopy (Renishaw system 1000; Renishaw plc, Gloucestershire, UK) with a backward scattering configuration. An Ar^+ laser (514.5 nm) was used as an excitation source. A laser beam was focused on the sample surface, and the spot size was approximately 1 μm. The measurement time was fixed at 1 min.

III. RESULTS AND DISCUSSION

A. La Content Dependence

All the La-PZT films with a Pb content ratio of 125% relative to a stoichiometric value were crystallized into single perovskite phase of the PZT system with rhombohedral or pseudo-cubic structure. The La-PZT films with La contents ranging from 0 to 9 mol% mostly had a 111 preferred orientation while the La-PZT films with La contents of 12 and 15 mol% had a random orientation. The La-PZT film with a La content of 0 mol% showed well-saturated and slim *D-E* hysteresis loop. Maximum polarization decreased and the squareness of the hysteresis loops monotonically deteriorated with increasing La content, and finally, ferroelectricity almost disappeared in the La-PZT films with La contents greater than 12 mol% as shown in Fig. 1. The decrease in maximum polarization seemed to be due to the decrease in the anisotropy of crystals by La substitution.

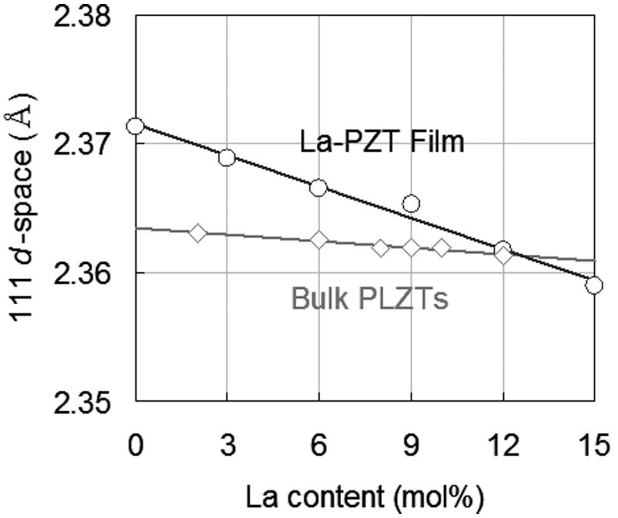

Fig. 2. The variation of the 111 *d*-space in bulk PLZTs [12] and La-PZT films with a Pb content ratio of 125% relative to a stoichiometric value as a function of La content.

Fig. 2 shows the variation of the 111 *d*-space in bulk PLZTs [12] and La-PZT films as a function of La content. The 111 *d*-space slightly decreases with increasing La content in bulk ceramics, although La ions have larger radius than Pb ions, which should be substituted. The La-PZT film with the La content of 0 mol% showed larger 111 *d*-space value than the bulk ceramics. This means that the La-PZT film had compressive stress from a substrate. The 111 *d*-space also decreased in the La-PZT film with increasing La content, but the decreasing rate in the film was obviously larger than that of the bulk ceramics. It seems that the lattice constant decreased with increasing La content in the La-PZT film as well as in bulk ceramics, and the decrease in lattice constant also caused relaxation

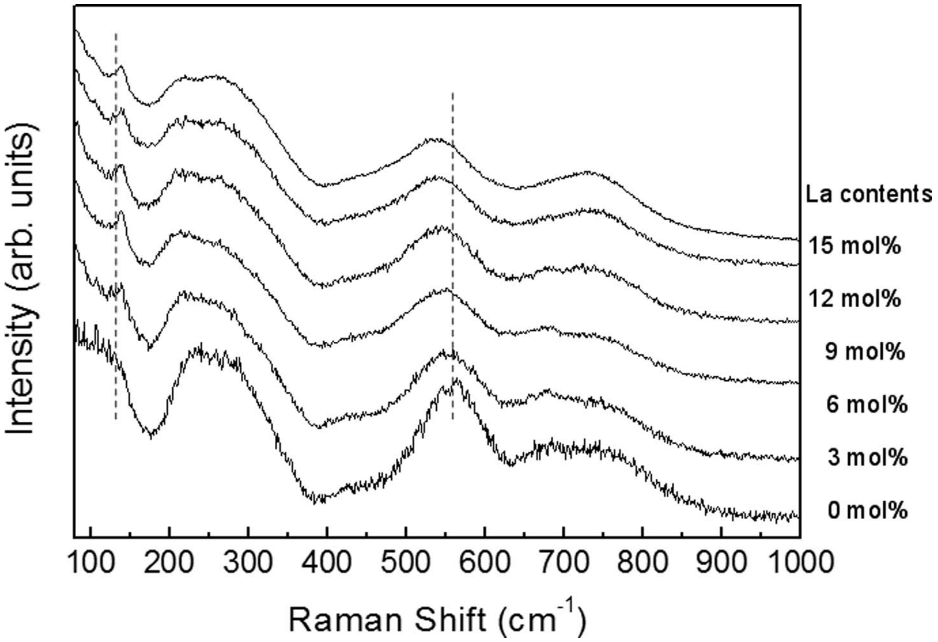

Fig. 3. The Raman spectra of La-PZT films with various La contents from 0 to 15 mol%.

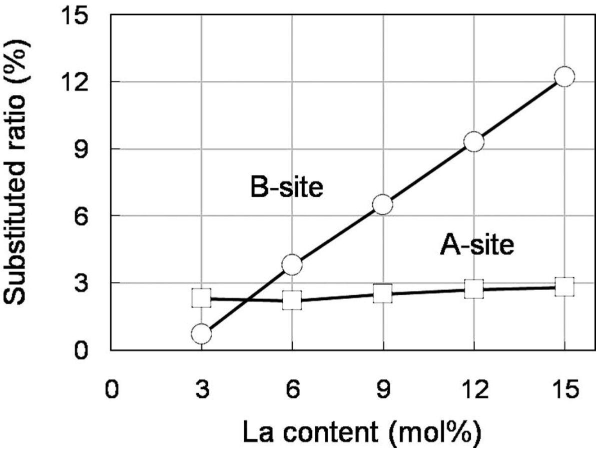

Fig. 4. The site occupancies estimated from the Raman spectra of La-PZT films with a Pb content ratio of 125% relative to a stoichiometric value as a function of La content.

of the compressive stress. This is a possible reason for the larger decreasing rate of 111 *d*-space in the La-PZT films.

Fig. 3 shows the Raman spectra of La-PZT films with La contents from 0 to 15 mol%. The peak at ~135 cm^{-1} corresponds to Pb (A-site in a perovskite structure, ABO_3)-based lattice mode [17], while that at ~560 cm^{-1} corresponds to the Zr/Ti-O (B site-O) stretching mode [18]. The site occupancies of La ions at the A-site were estimated from the peak at ~135 cm^{-1}, while that of La ions at the B-site were estimated from the peak at ~560 cm^{-1}. Details of the calculation method for the site occupancy were described elsewhere [19], [20]. Fig. 4 shows the site occupancies estimated from the Raman spectra. From Fig. 4, it is found that La ions were also substituted for B-site ions at La contents greater than 3 mol%, while La ions were selectively substituted only for A-site ions up to 3 mol% in the La-PZT films. There is a possibility that quadrivalent Pb ions occupy the B-site and affect electrical behavior [21], [22]. Unfortunately, we cannot say for certain whether quadrivalent Pb ions occupied the B-site or not in the La-PZT film with the La content of 0 mol%. However, it is obvious that La ions were mainly substituted for the A-site and further substitution of quadrivalent Pb ions for the B-site rarely occurred in the case of the La content of 3 mol% because the peak shifts at ~135 cm^{-1} were larger than at ~560 cm^{-1}, and the summation of estimated substituted values was almost 3 mol%. Furthermore, we considered that only the La ions were substituted for the B-site above the La content of 3 mol% because only the peak at ~560 cm^{-1} shifted with increasing the La content while the peak at ~135 cm^{-1} did not move.

From these results, we concluded that, in the CSD-derived La-PZT films prepared from precursor solutions with excess Pb element of 125% relative to a stoichiometric values, La ions were selectively substituted for A-site ions up to approximately 3 mol% and caused the reduction of lattice constants and anisotropy, and La ions greater than 3 mol% were substituted for B-site ions, although they still caused the reduction of lattice constants and anisotropy.

B. Pb Content Dependence

Fig. 5 shows the site occupancy of La ions in the La-PZT films with an La content of 3 mol% as a function of Pb content. At Pb contents of 107 and 112 mol%, accurate La-substituted ratios were not able to be estimated because of many Pb vacancies. At Pb contents of 116 and 121 mol%, the total substituted ratios for A-site and B-site were almost 3 mol%. Therefore, it seems that almost

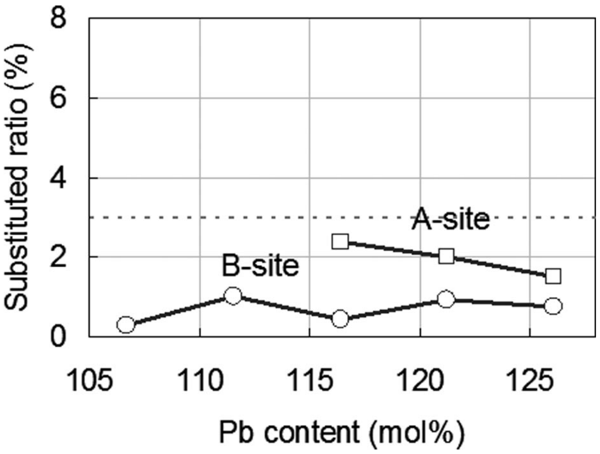

Fig. 5. The site occupancies estimated from the Raman spectra of La-PZT films with an La content of 3 mol% as a function of Pb content.

all La ions were substituted for A-site or B-site, and there were few Pb vacancies in this region. The La-substituted ratio for A-site was slightly decreased with increasing Pb content from 116 to 121 mol%. This suggests that excess Pb prevented the A-site substitution of La ions. At the Pb content of 126 mol%, the total substituted ratio was 2.28 mol%, less than 3 mol%. This result suggests that some of the La ions were not substituted either for A-site or B-site ions because of too much Pb.

Fig. 6 shows the variation of the 111 *d*-space in La-PZT films as a function of Pb content. The 111 *d*-space slightly increased with increasing Pb content from 107 to 112 mol%. This seems to be caused by decreasing Pb vacancies. The 111 *d*-space were almost constant at Pb contents ranging from 112 to 121 mol%. On the other hand, the 111 *d*-space obviously increased with increasing Pb content from 121 to 126 mol%. It seems that La and excess Pb ions interstitially existed in the crystals and expanded the unit cell.

All the La-PZT films with an La content of 3 mol% and various Pb contents showed well-saturated *D*-*E* hysteresis loops similar to Fig. 1(b). The values of remanent polarization were almost constant, although only the La-PZT film with a Pb content of 107 mol% showed slightly lower remanent polarization. The existence of Pb vacancies seems to be the reason for the lower remanent polarization.

From these results, we concluded that, in the La-PZT films with a La content of 3 mol%, the Pb content of 116 mol% (120% relative to a stoichiometric value) was optimum from the viewpoint of site occupancy. However, we previously reported that surface morphology of the La-PZT films with a Pb content of 116 mol% was not a well-filled structure [10]. The control of the surface morphology at the optimum composition for the site occupation will be the subject of future work.

IV. CONCLUSIONS

Polycrystalline La-PZT films with various La and Pb contents were fabricated on Pt/Ti/SiO_2/Si substrates by CSD. The 111 preferred-oriented La-PZT films were obtained at La contents ranging from 0 to 9 mol%, and randomly oriented films were obtained at La contents greater than 12 mol%. We found that in the La-PZT films with a Pb content ratio of 125% relative to a stoichiometric value, La ions were substituted not only for A-site ions but also for B-site ions at La contents greater than 3 mol%. Although the influence of La substitution for B-site on electro-optic properties is not yet clear, La substitution for the B-site seems to cause larger reduction of the unit cell size. In the La-PZT films with an La content of 3 mol%, below Pb contents of 116 mol%, there were Pb vacancies in the films while the Pb content greater than 121 mol%, La and excess Pb ions interstitially existed in the crystals and expanded the unit cell. These results suggest that excess Pb prevented the A-site substitution of La ions. Finally, we concluded that in the La-PZT films with a La content of 3 mol%, the Pb content of 116 mol% (120% relative to a stoichiometric value) was optimum from the viewpoint of site occupancy.

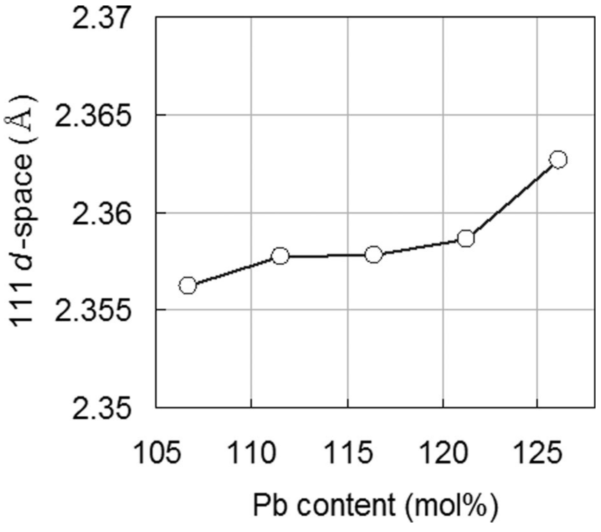

Fig. 6. The variation of the 111 *d*-space in La-PZT films with a La content of 3 mol% as a function of Pb content.

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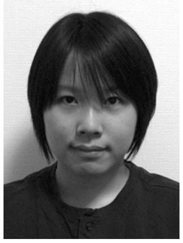

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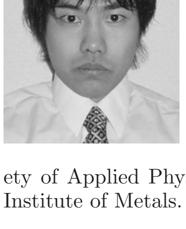

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