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Upconversion photoluminescence of epitaxial Yb^{3+}/Er^{3+} codoped ferroelectric $Pb(Zr,Ti)O_3$ films on silicon substrates

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ABSTRACT

Thin films of Yb^{3+}/Er^{3+} codoped $Pb(Zr,Ti)O_3$ (PZT:Yb/Er) have been epitaxially grown on the $SrTiO_3$ buffered Si wafer by pulsed laser deposition. Strong upconversion photoluminescence was observed in the PZT:Yb/Er thin film. Using piezoresponse force microscopy, polar domains in the PZT:Yb/Er film can be reversibly switched with a phase change of 180°. Ferroelectric hysteresis loop shape with a well-saturated response was observed. The epitaxially grown lanthanide-doped PZT on silicon opens up a promising route to the integration of luminescent functional oxides on the silicon platform.

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1. Introduction

In recent years, photoluminescence (PL) properties originating from lanthanide doped ferroelectric materials have attracted significant interests due to their promising potential for multifunctional optoelectronic applications [1–5]. Trivalent lanthanide (Ln^{3+}) ions, such as Eu^{3+}, Tm^{3+}, Pr^{3+}, and Er^{3+}, can be incorporated into different systems as activators to acquire outstanding luminescence properties [6–10]. Widely studied ferroelectric materials include $BaTiO_3$ and $Pb(Zr,Ti)O_3$ (PZT) with typical perovskite ABO_3 structure. These highly functional perovskite oxides possess robust crystal structures and low vibrational frequency, which make them suitable for hosting Ln^{3+} ions [11–14]. The integration of ferroelectricity, piezoelectricity and photoexcitation based on Ln^{3+}-doped ferroelectrics provides great flexibility and inspiration to fabricate multifunctional and all-in-one devices [15], *e.g.* a micro-opto-electro-mechanical system. The performance of such devices can be further improved by using thin film layers with, for example, the operating voltages being dramatically reduced. Monolithic device integration will then be possible on various platforms, including with CMOS or integrated photonics circuits. Along this direction, Ln^{3+} doped luminescent thin films have been extensively studied in the optoelectronics community [16–18]. Part of us have reported *in-situ* and real-time tunable luminescence of Ln^{3+} doped ferroelectric thin films *via* electric field, as well as strain fields [19–21]. The material system meets the requirement for tuning the light emission of Ln^{3+} ions and is distinctly different to other more conventional chemical approaches [5].

Integrating Ln^{3+} doped luminescent ferroelectric thin film on silicon may open up many exciting possibilities. Silicon-based optoelectronic devices play a profound role in the era of "system-on-a-chip" due to their excellent compatibility with sophisticated integrated circuit technology. Many of the envisioned applications require the miniaturization and integration of the light sources on chip [22]. With further incorporation of ferroelectric properties, the luminescent (*e.g.* Ln^{3+} doped) thin film on Si may lead to a multifunctional on chip integrated device. There have been considerable efforts of growing Ln^{3+} doped ferroelectric thin film on Si substrates, however, in most cases the thin films are polycrystalline [23,24]. This is a significant problem for potential applications, since the polycrystalline films in general exhibit properties, including piezoelectric coefficients, polarization and dielectric constant, inferior to those of their epitaxial counterparts [25,26]. As Si has a high reactivity with oxygen and large lattice mismatch with most perovskite oxides, the direct integration of high-quality epitaxial Ln^{3+} doped ferroelectric thin films on Si is still challenging.

In this work, we deposited epitaxial Yb^{3+}/Er^{3+} codoped PZT on Si by pulsed laser deposition (PLD). A thin layer of $SrTiO_3$ (STO) is introduced

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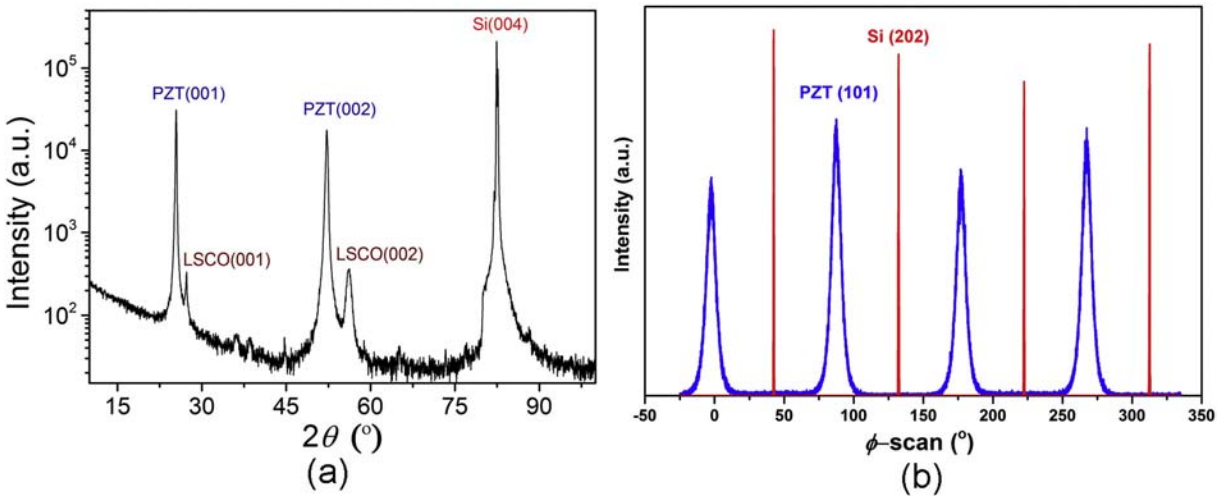

Fig. 1. (a) XRD θ–2θ scan of the PZT:Yb/Er/LSCO/STO heterostructure grown on Si. (b) ϕ-Scans of PZT (101) and Si (202), showing the 45° rotated cube-on-cube epitaxy.

to provide a template for incorporating epitaxial oxide heterostructures on Si [27–29]. The STO buffer layer acts simultaneously as structural template and barrier for cation migration. We investigated the coexistence of luminescence and ferroelectricity in the epitaxial Ln^{3+} doped PZT thin films grown on STO buffered Si wafer. Strong upconversion emission is observed. Piezoresponse force microscopy (PFM) experiments show that the ferroelectric domains can be written and read, and they can be switched reversibly by changing the applied bias. We envision that these high quality epitaxial luminescent ferroelectric films will play an important role in multifunctional Si-based micro-opto-electro-mechanical systems.

2. Experimental details

The STO buffer layer was prepared in a molecular beam epitaxy chamber with a base pressure of 5×10^{-10} mbar. A 4-nm-thick STO film was deposited on a high-resistive, 500-μm-thick Si (001) substrate using the process as described in ref. [29]. Yb^{3+}/Er^{3+} codoped PZT (PZT:Yb/Er) target with a chemical formula $Pb_{1.1}(Zr_{0.52}Ti_{0.45}Yb_{0.025}Er_{0.005})O_3$ was prepared using standard solid state reaction. PZT(Yb/Er) target contains 10 mol% excess PbO to prevent Pb deficiency caused by the high Pb volatility. The films were grown by PLD with a KrF excimer laser at the wavelength of 248 nm. A 20 nm thick lattice matched $La_{0.7}Sr_{0.3}CoO_3$ (LSCO) was used as the bottom electrode between the PZT:Yb/Er film and the STO buffered Si substrate. The LSCO layer was deposited with the substrate temperature of 575 °C, oxygen pressure of 0.1 mbar and a laser pulse energy density of 2 J/cm^2. The PZT:Yb/Er film with a thickness of 40 nm was grown with a frequency of 2 Hz and a laser pulse energy density of 2 J/cm^2. The grown temperature and oxygen pressure were fixed at 575 °C and 0.2 mbar, respectively. After the deposition, the films were post-annealed *in situ* at the growth temperature in 0.5 atm oxygen pressure for 30 min before they were cooled down to room temperature.

Structural properties of the oxide stack on silicon were investigated by an X-ray diffractometer (XRD) in the θ–2θ geometry with a Bruker D8 Advance with Co-K_α radiation. The ϕ scans were performed with a texture goniometer Phillips X'pert equipped with Cu-K_α radiation. The PL spectra were recorded using a spectrometer under 980 nm laser excitation. The surface morphology, local ferroelectric properties were measured using an Asylum Research Cypher scanning probe microscope. All measurements were performed at room temperature.

3. Results and discussion

Fig. 1 shows the XRD patterns of the PZT:Yb/Er/LSCO/STO heterostructure grown on Si. In the θ–2θ scan, only the (00l) peaks of PZT are presented in the diffraction pattern, indicating that the PZT along with the LSCO layer has a *c*-axis oriented growth on the STO buffered Si substrate. The characteristic diffraction peaks of PZT can be observed without any secondary phase or random orientations, suggesting that the Yb^{3+} and Er^{3+} ions were efficiently doped into the PZT host lattice. There is no apparent peak corresponding to STO in Fig. 1a due to the small thickness of the STO buffer layer. To further evaluate the in-plane alignment of the film, the ϕ scans of the (101) reflection were performed and the results are shown in Fig. 1b. The epitaxial relationship between the oxide layers and the substrate can be confirmed: PZT:Yb/Er[100]||LSCO[100]||STO[100]||Si[110]. The ϕ scans also indicate that the epitaxial oxide stack has been grown with a 45° rotation with respect to the Si unit cell.

Fig. 2 shows the upconversion spectrum of a PZT:Yb/Er thin film grown on STO buffered Si wafer under 980 nm laser excitation. The PZT:Yb/Er film exhibits two strong green bands located at 523 and 550 nm corresponding to the $2H_{11/2}/4S_{3/2} \rightarrow 4I_{15/2}$ transitions, and a weak red emission band at 656 nm corresponding to the $^4F_{9/2} \rightarrow {}^4I_{15/2}$ transition of Er^{3+} ion, respectively. The corresponding color point (0.28, 0.68) in the 1931 Commission International de l'Eclairage (CIE) chromaticity diagram is shown in the inset of Fig. 2. For better understanding of the upconversion processes, Fig. 3 shows the energy level

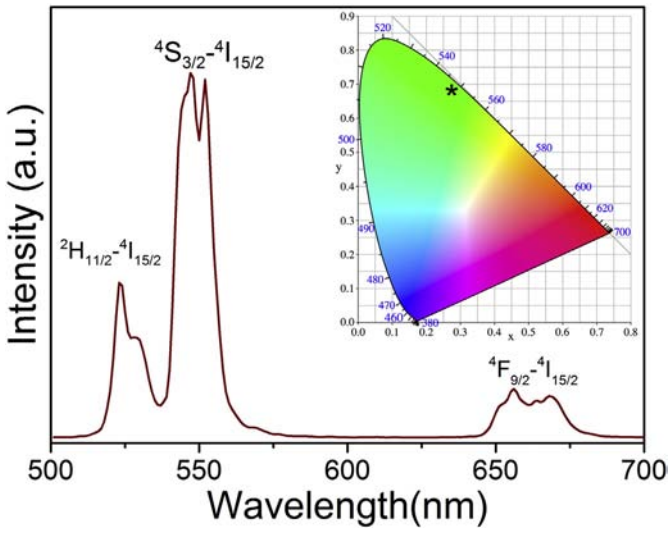

Fig. 2. The upconversion emission spectra of the PZT:Yb/Er/LSCO/STO/Si structure under 980 nm laser excitation. The inset shows the CIE chromaticity diagram for PZT:Yb/Er.

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