



# Growth and characterization of periodically polarity-inverted ZnO structures on sapphire substrates

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## ABSTRACT

We report on the fabrication and characterization of periodically polarity inverted (PPI) ZnO heterostructures on (0 0 0 1) Al<sub>2</sub>O<sub>3</sub> substrates. For the periodically inverted array of ZnO polarity, CrN and Cr<sub>2</sub>O<sub>3</sub> polarity selection buffer layers are used for the Zn- and O-polar ZnO films, respectively. The change of polarity and period in fabricated ZnO structures is evaluated by diffraction patterns and polarity sensitive piezo-response microscopy. Finally, PPI ZnO structures with subnanometer scale period are demonstrated by using holographic lithography and regrowth techniques.

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## 1. Introduction

ZnO has attracted much interesting as an optoelectronic and nonlinear optical material by using its high exciton binding energy and second order susceptibility [1–3]. However, due to the unstable performance of p-type ZnO material, many researchers are looking for the other application fields. In the case of nonlinear optical devices, there has been continuous demand for novel nonlinear optical materials instead of bulk materials such as LiNbO<sub>3</sub> and LiTaO<sub>3</sub> for potential applications in integrated optics. Previously suggested these promising bulk materials have drawback for the cost and flexibility. Nanostructure materials have many novel properties due to size effect, which can be used to solve some bulk materials problems [4,5]. Hence we want to propose the periodically polarity-inverted (PPI) ZnO thin film structures to overcome the current problems coming from using of bulk material which will open new application fields of ZnO in nonlinear optical devices as like the periodically poled GaN [6].

To date, however, only a few results have been reported on the fabrication of periodically polarity changed ZnO structures on sapphire substrates [7,8]. It was be so, but the optical and structural study on the PPI ZnO structures is considered as an indispensable step toward the various applications such as

electronic, optoelectronic, electrochemical, and electromechanical devices [9,10].

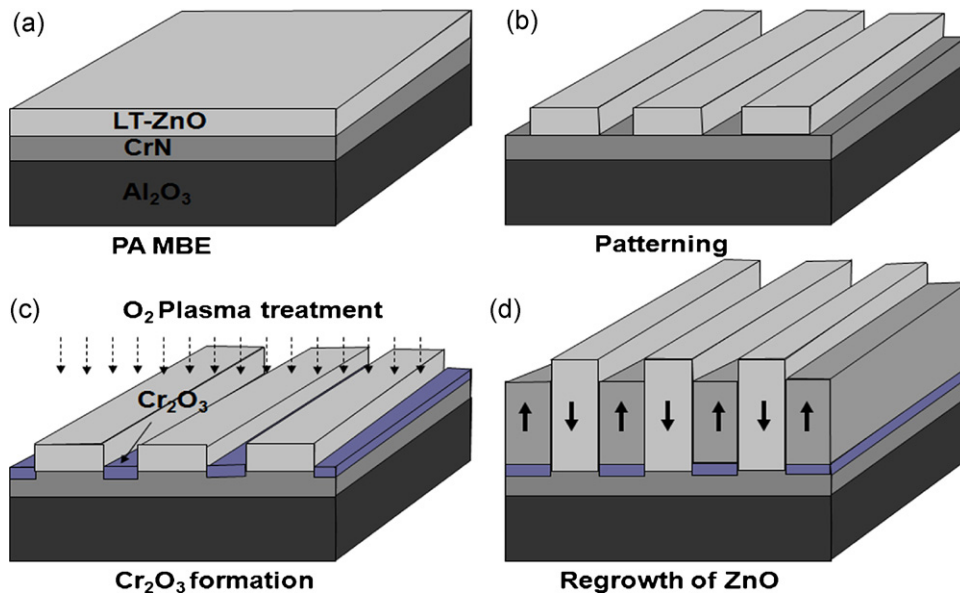
In this article, we report on the successful fabrication of nanometer scale PPI ZnO structures on (0 0 0 1) Al<sub>2</sub>O<sub>3</sub> substrate by plasma assisted molecular beam epitaxy (PA MBE) with holographic lithography. The employment of Cr compound layers (CrN and Cr<sub>2</sub>O<sub>3</sub>) as a buffer layer for ZnO film makes it possible to select the polarity of ZnO as Zn- and O-polarity, respectively [11]. In here, we used the Cr<sub>2</sub>O<sub>3</sub> layer replace with Al<sub>2</sub>O<sub>3</sub> for high quality O-polar ZnO without the rotational domain. The advantages of Cr<sub>2</sub>O<sub>3</sub> buffer layer comparing to sapphire substrates for ZnO growth can be found in other article [12].

## 2. Experimental

Fig. 1 shows a schematic procedure to make the PPI ZnO heterostructure on c-sapphire. After the growth of low temperature (LT) ZnO on Zn exposed CrN buffer layer [7], stripe patterns were formed by chemical or physical etching method and with holographic lithography using the conventional photoresist-patterned mask with 0.5–30 μm-width stripes. Selective etching was conducted to remove the LT ZnO/CrN till the CrN buffers. After transfer templates into the MBE chamber, the O<sub>2</sub> plasma treatment was conducted at 650 °C for 10 min to make the Cr<sub>2</sub>O<sub>3</sub> on opened CrN surface. The formed Cr<sub>2</sub>O<sub>3</sub> layers show the single crystalline thin films structures [11]. Finally, ZnO film growth was started on the patterned templates, which initiated the growth of ZnO on the

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**Fig. 1.** Schematic illustration of overall processes to fabricate the PPI ZnO structure; (a) the growth of LT ZnO on CrN, (b) 1D striping patterning using the chemical or holographic lithography, (c) Cr<sub>2</sub>O<sub>3</sub> formation by O<sub>2</sub> plasma treatment, (d) HT ZnO growth to fabrication of PPI ZnO.

CrN and Cr<sub>2</sub>O<sub>3</sub> regions at the same time. Here, note that the ZnO films on CrN have Zn-polarity and the films on Cr<sub>2</sub>O<sub>3</sub> have O-polarity [11]. Therefore, the final structure shown in Fig. 1 should confirm a periodical polarity-inverted structures consisted with Zn- and O-polar ZnO films in lateral direction on a same substrates.

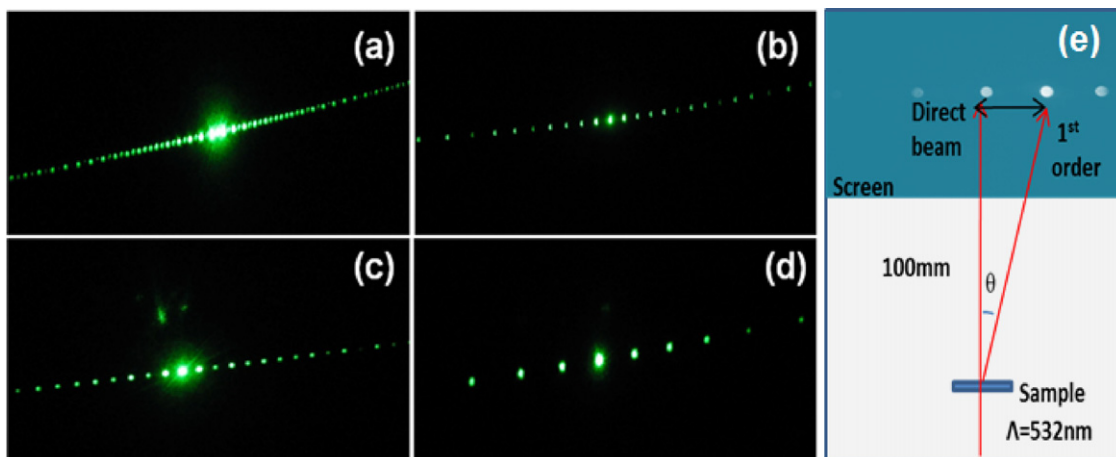
The success of periodical polarity control was confirmed by means of surface piezo-voltage difference by using a piezo-response microscopy (PRM) images which certified the periodical polarity inversion. The period was evaluated by both optical microscopy and diffraction pattern analysis from 532 nm green laser. High resolution transmission electron microscopy (HR TEM) and micro photoluminescence ( $\mu$ -PL) are used to investigate structural and optical properties of the PPI structure, respectively.

### 3. Results and discussion

#### 3.1. Evaluation of lateral polarity inversion and period

In order to evaluate the period of fabricated PPI ZnO, diffraction pattern analyses were performed. Fig. 2 shows the

change of diffraction pattern depending on a periodicity in 1D PPI ZnO structures. The distance between sample and screen is fixed as 10 mm. Here, one period is defined as the length for the one pair of Zn- and O-polar films. The periodicity of PPI structures can be estimated easily by Bragg's diffraction equation;  $\lambda = d \times \sin \theta$  (here,  $\theta$ : the angle between transmission spot and 1st order diffracted spot,  $\lambda$ : wave length of laser source, and  $d$ : periodicity) [13]. Fig. 2(e) shows the experimental set-up and periodicity calculation method. From the distance between direct laser beam and 1st diffracted beam, we can calculate the real periodicity of sample. The estimated values of periodicity from the measurement results are well agreed with the real periodicity obtained from the optical microscopy as shown in Fig. 3(a). Moreover, the determination of lateral polarity inversion has been conducted by PRM technique. The image of piezoelectric response for the fabricated structure clearly showed the different brightness as shown in Fig. 3(b) and revealed the periodical change of response voltage (phase) by applied AC voltage. The details for the PRM usage for the polarity determination can be found out other Ref. [7].



**Fig. 2.** Diffraction pattern change as a function of periodicity in PPI ZnO with (a) 60, (b) 20, (c) 10, and (d) 4  $\mu$ m. (e) The experimental set-up for the analysis of diffraction patterns.

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