



# Morphological, Optical, and Crystalline Analysis of ZnTiO<sub>3</sub> Nanostructures Deposited on Porous Silicon Substrate

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## Abstract

Zinc titanate (ZnTiO<sub>3</sub>) was grown on silicon and porous silicon. ZnTiO<sub>3</sub> layers were prepared by sol-gel method. Porous silicon was fabricated by electrochemical etching of silicon in HF solution. The effect of substrate porosity on morphology, structure and optical properties of ZnTiO<sub>3</sub> nanostructures has been studied. These properties were investigated using XRD, Ultraviolet–Visible spectroscopy, and HRTEM. Some important parameters (absorption, reflectivity (R (%)) and grain size) were studied. It was found that the Structural, morphology and optical properties of ZnTiO<sub>3</sub> layers are dependent strongly on the type of substrates. The crystalline size decreased for ZnTiO<sub>3</sub> layers deposited on PS substrate. The average grain size is about 80 nm for ZnTiO<sub>3</sub> grown on porous silicon. The surface morphology of films was also found to be uniform and homogeneous. ZnTiO<sub>3</sub>-PS shows enhancing photon absorption compared to ZnTiO<sub>3</sub>-Si.

**Keywords** ZnTiO<sub>3</sub> · Porous silicon · UV-Vis · XRD · HRTEM

## 1 Introduction

Metal oxide semiconductors have recently shown great influence in the fields of photocatalysis [1–3], optoelectronics [4–6], and solar cells [7]. Amongst the studied semiconductors, Zinc oxide (ZnO) has received much attention. It is a wide-bandgap oxide semiconductor with a direct energy gap of about 3.37 eV. ZnO has high chemical and mechanical stability; furthermore, it is nontoxic and widespread in nature [8, 9]. TiO<sub>2</sub> has also shown promise in the areas of photocatalysis [2], solar cells [10], gas sensor and other optical applications [11, 12]. Enormous increase of applications based on ZnO and TiO<sub>2</sub> has been caused not only by the improvements

of their intrinsic properties but also by the achievements of transition metal doping and mixed oxides formations.

So, TiO<sub>2</sub> and ZnO have been used to fabricate ZnO-TiO<sub>2</sub> composite materials, annealing conditions and ZnO/TiO<sub>2</sub> molar ratio were found have significant effects on the phase formation [13]. The coupling TiO<sub>2</sub>/ZnO gives a material named zinc titanate that enhances the properties of ZnO and TiO<sub>2</sub>, for example, by widening its light absorption spectrum [14]. Additionally, the photocatalytic activity of oxides may help reduce the susceptibility of pollutants to form aggregate structures [15].

However, zinc titanate system has three compounds that exist in the: ZnTiO<sub>3</sub>, Zn<sub>2</sub>Ti<sub>3</sub>O<sub>8</sub>, and Zn<sub>2</sub>TiO<sub>4</sub>, which it remains as a structural defect. ZnTiO<sub>3</sub> is a perovskite-type oxide (ABO<sub>3</sub>) whose perovskite structure endows the flexibility to alter the arrangement of the A- and B-sites, and incorporates cation combinations at the A- and B-sites to assemble substituted perovskites. ABO<sub>3</sub> have been considered as materials with various applications in solid oxide fuel cell electrodes (SOFC) [16], metal barriers [17], sensors [18], electronics [19] and catalysts [20]. As a well-known member of this family, zinc titanate (ZnTiO<sub>3</sub>) has been used as pigments [21], dye adsorbents [22], sensors such as NO and CO gases [23], microwave resonator materials [24], heat reflective pigments [25] and Photocatalyst [26].

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Several methods are used to synthesize Zinc titanate ( $\text{ZnTiO}_3$ ), as solid state reaction [27], Sol-gel [28, 29], Pechini process [30], Hydrothermal method [31], Sputtering [32], Microwave heating [33], and molten salt Method [34]. Zinc titanate ( $\text{ZnTiO}_3$ ) thin films deposited on different substrates. The investigation of the influence of various substrates such as Si (100) [35], glass [36], quartz substrate [37], and ITO coated glass [38] has also been studied.

In This work, porous silicon (PS) is another candidate as substrates due to adjustable roughness (large internal surface), high resistance, strong absorbability, and potential for the development of silicon-based optoelectronic devices. Porous silicon has been used to deposit metal oxides and obtain good quality thin layers [39–41]. we report the growth of  $\text{ZnTiO}_3$  hexagonal and nanoscale on silicon and porous silicon substrates by spin coating. Optical, morphology and crystal structure of hexagonal and nanoscale were studied by UV-Vis, XRD and TEM techniques. The experimental findings on the effect of the sponge-like structure of the porous silicon substrates on the crystallinity properties of the  $\text{ZnTiO}_3$  thin films are presented and the causes are discussed.

## 2 Experimental Details

For the fabrication of nanostructures, porous Si (PSi) substrates were obtained by electrochemical etching [40, 41] of p-type, (100) oriented silicon (Si) wafers with a resistivity of 10–20  $\Omega$  cm. For the preparation of the porous substrates, c-Si wafers were cleaned with 2-propanol (under sonication) for 4 min. The Si wafers were rinsed with deionized water, dried with  $\text{N}_2$  flux, and immersed in hydrofluoric acid aqueous solution (2%) for 4 min, followed by rinsing with deionized water and ethanol and dried under  $\text{N}_2$  flux. The etching process was carried out for 15 min in hydrofluoric acid (HF, 40 wt%) and ethanol (1:1 volumetric ratio) solution at a constant current density of 10 mA/cm<sup>2</sup>. After the etching process, the substrates were rinsed with ethanol and dried with  $\text{N}_2$  flux.

On the other hand,  $\text{ZnTiO}_3$  nanostructured films on PSi and Si substrates were obtained by spin coating deposition. The formation of  $\text{ZnTiO}_3$  and the corresponding characterization have been widely studied in the literature [28, 29, 35]. Zinc titanate thin films was prepared by the sol-gel method. In general, Zinc acetate dihydrate ( $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ , Sigma Aldrich, reagent grade, 99% purity), and Titanium (IV) n-butoxide ( $\text{Ti}(\text{O}(\text{CH}_2)_3\text{CH}_3)_4$ , Sigma Aldrich, reagent grade, 99% purity) were used as Zn and Ti source materials, respectively. The Zinc acetate and tetrabutyl titanate were dissolved into ethylene glycol monoethyl ether and acid acetic acid solvent at 80 °C and stirred for 30 min to form clear solution.

For the deposition, PSi and Si substrates with a dimension of 20 mm × 20 mm were used. The substrates were washed successively with acetone, hydrochloric acid aqueous solution, deionized water and absolute ethanol in an ultrasound bath. Then, dried at 100 °C for 10 min before coating.  $\text{ZnTiO}_3$  precursor solution was spin-coated on the substrates at 3000 rpm during 30 sec. The as-prepared films were annealed at 120 °C for 10 min to remove organic materials and then at 700 °C for 2 hours to crystallize them into a perovskite structure in a rapid thermal annealing furnace. The process from coating to annealing was repeated 2, 4, 6 and 8 times to produce different thickness of the films. The as prepared and sintered samples were subjected to various analyses by suitable analytical technique.

The structural properties of the prepared materials were studied by XRD analysis using (BRUKER D8 advance model, at room temperature). The morphology of  $\text{ZnTiO}_3$  was analyzed by high resolution transmission microscopy (TEM), using a HRTEM JEOL2100F microscope. UV-vis absorbance and reflectance spectroscopy analysis (UV-vis) was carried out using Perkin Elmer Lambda 950 spectrophotometer.

## 3 Result and Discussion

### 3.1 XRD Analysis

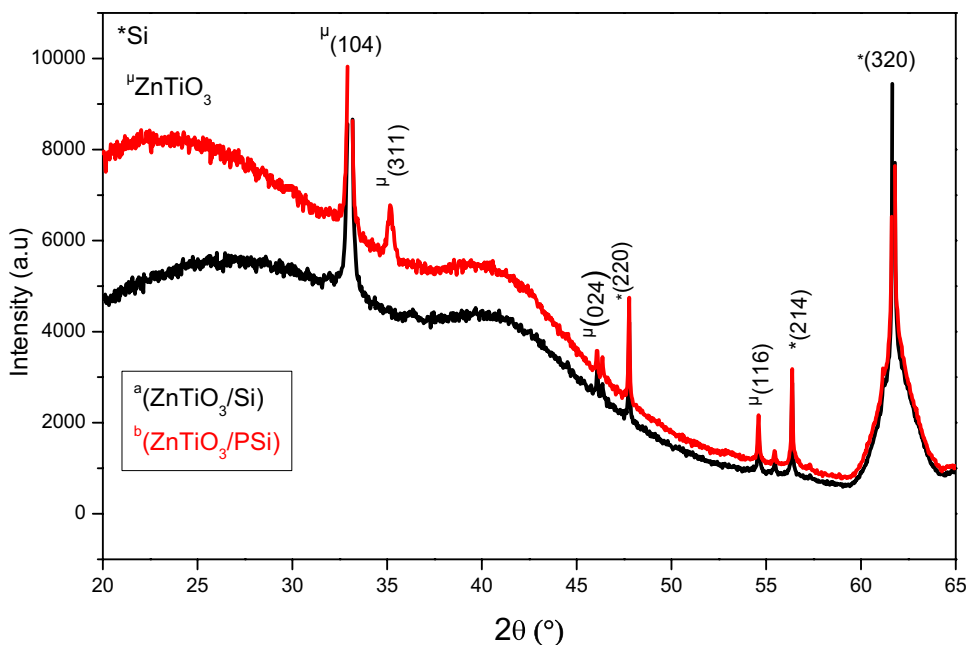
The thicknesses of the films resulting of the 2, 4, 6 and 8 repetitions of spin coating and annealing were found to be 80 nm, 120 nm, 160 nm and 240 nm.

To study the effect of substrate (Silicon, Porous silicon) on crystal structure of  $\text{ZnTiO}_3$ , we performed XRD experiments on the samples. The XRD analysis of  $\text{ZnTiO}_3/\text{Si}$  and  $\text{ZnTiO}_3/\text{PSi}$  were reported as in Fig. 1. The crystal planes (104), (110), (024), (116), (214), for hexagonal  $\text{ZnTiO}_3$  (JCPDS card No.26–1500) in the diffractograms can be indexed [30, 42, 43].

The (104) peak is more intense in the  $\text{ZnTiO}_3/\text{PSi}$  compare to  $\text{ZnO}/\text{Si}$ . The peak (110) peak appeared in the  $\text{ZnTiO}_3/\text{PSi}$  compare to  $\text{ZnO}/\text{Si}$ . Consequently, there traduces better crystallinity of  $\text{ZnTiO}_3$  formed on PSi than on Si. The difference in intensity can be explained by higher absorption rate of the capillary effect presented in  $\text{ZnTiO}_3/\text{PSi}$  sample and the high adhesion due to the high specific surface area in the case of PSi.

The average crystallite sizes of the nanoparticles  $\text{ZnTiO}_3$  can be estimated using Scherrer equation which is defined as  $d = (0.94 k) / (b \cos(\theta))$ , where  $d$  is the average grain size,  $k$  is the X-ray wavelength (0.15406 nm),  $b$  is the full-width at half maximum (FWHM), and  $\theta$  is the diffraction angle (32.9°). The strongest peaks (104) in XRD were used to calculate the grain size for  $\text{ZnTiO}_3$ . Applying Scherrer's

**Fig. 1** X-ray diffraction of ZnTiO<sub>3</sub> nanostructures grown on conditions on (a) Si substrate, (b) PSi substrate

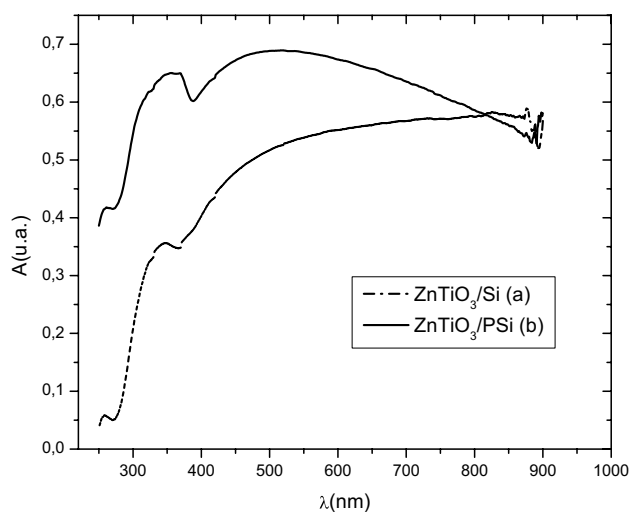


formula, the grain sizes were found to be 45 nm and 20 nm for ZnTiO<sub>3</sub> formed on PSi and ZnTiO<sub>3</sub> formed on Si, respectively. The obtained values indicate that the porous layer has a significant effect on the synthesis mechanism of ZnTiO<sub>3</sub> nanostructures and can serve as the starting point for the growth of nanostructures. Therefore, the rough surface morphology of PSi plays a major role in controlling the growth of the wetting layer [44]. Due to its special surface morphology, the porous layer is a good substrate for lattice-mismatched heteroepitaxy. The surface of the porous silicon layer is composed of many nanocrystals. These Si nanocrystals maintain (100) orientation with the outer surface of the silicon wafer. These randomly distributed Si crystallites on the surface act as nucleation sites and induce the growth of ZnTiO<sub>3</sub> nanostructures along the preferred orientation.

### 3.2 Optical Properties

Optical properties of the ZnTiO<sub>3</sub>/Si and ZnTiO<sub>3</sub>/PSi samples have been investigated using UV-Vis spectroscopy. Figure 2 compares the absorbance of ZnTiO<sub>3</sub>/Si and ZnTiO<sub>3</sub>/PSi. The layer thickness of ZnTiO<sub>3</sub> for two samples is on the order of 120 nm. As shown in Fig. 2, the absorbance recorded in the spectral range 250–900 nm. The spectrum a corresponding to the samples reveals three bands. A first UV absorption band extending from 250 to 270 nm with a sharp band located at about 260 nm, second band with a centered at about 350 nm, and three absorption band extending from 350 to 900 nm.

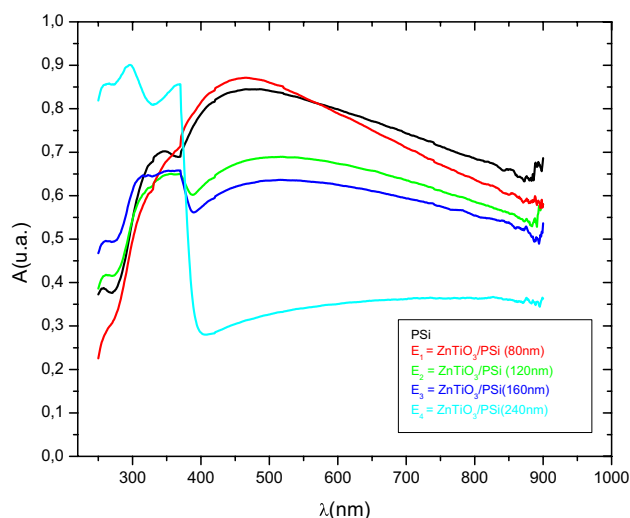
The band edge is observed at ~280 (first band) and ~400 (second band) nm for all samples. Those bands edge absorption intensity show its absorbance capacity in UV light of



**Fig. 2** UV-Vis spectra of ZnTiO<sub>3</sub> nanostructures grown on conditions on (a) Si substrate, (b) PSi substrate

ZnTiO<sub>3</sub>, it is similar as reported in the literature [45, 46]. The absorbance increases with the porous layer due to the increasing to optical path in porous silicon specific surface. The increase in the visible absorbance (three band) confirmed that the ZnTiO<sub>3</sub> nanoparticles are emerged in the pores and therefore are deposited on the specific surface. We deduce that the ZnTiO<sub>3</sub>/PS acts as an efficient solar absorber.

Optical absorbance spectra of multilayer ZnTiO<sub>3</sub> films on porous silicon are shown in Fig. 3. It is seen from this Figure; the spectrum is similar to Fig. 2 with variation of absorbance intensity. The results show the strongest absorbance in the UV region of 250–400 nm increase with layer



**Fig. 3** UV-Vis spectra of ZnTiO<sub>3</sub> nanostructures with different thickness grown on PSi substrate

thickness of ZnTiO<sub>3</sub>. Figure 3 shows the absorption intensity increase of E<sub>1</sub>, compared to porous silicon and the other samples. The enhanced absorption of ZnTiO<sub>3</sub> due to the Surface Plasmon Resonance (SPR) of the free electrons and ZnTiO<sub>3</sub> incorporated in pores.

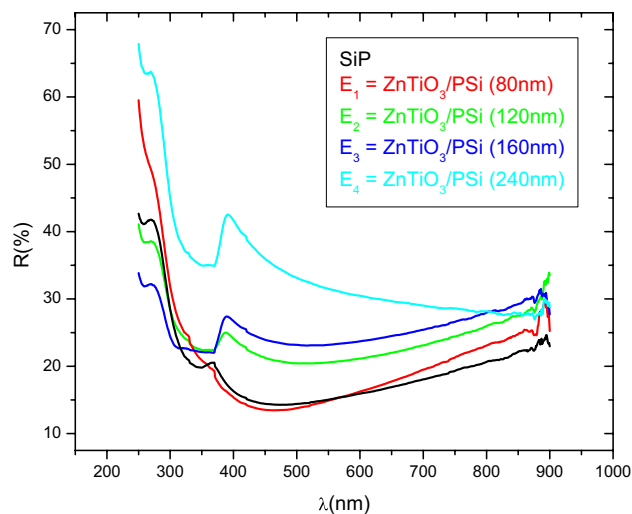
Figure 3 shows the absorption intensity in visible region decrease with layer thickness of ZnTiO<sub>3</sub>. It is because the layer become thick, getting larger and smoother, causing a reduction in the absorption optical.

The sample E<sub>4</sub> has high UV absorbance, which indicates this sample has synergistically enhanced UV absorption behavior. This is very beneficial for enhanced anti-UV aging performance. These conclusions are consistent with the statistical results as shown in Fig. 4.

Figure 4 shows UV–vis reflectance spectra of PS (black line), E<sub>1</sub> (red line), E<sub>2</sub> (green line), E<sub>3</sub> (blue line) and E<sub>4</sub> (light blue line) structures for the wavelength range of 250 nm–900 nm. The reflectivity of the porous silicon surface without ZnTiO<sub>3</sub> was around 14.3% and decreased to around 13.4% after layer of ZnTiO<sub>3</sub> deposited on porous silicon at around 450 nm. This is due to the formation of needle-like structures that result in enhanced light trapping. The decreased reflectivity results in significant increase of absorbance in E<sub>1</sub>. It can be observed from the spectra that the optical reflectance spectra of nZnTiO<sub>3</sub>/PS increased significantly with thickness layer. The increased reflectance results in significant decrease of absorbance in E<sub>2</sub>, E<sub>3</sub> and E<sub>4</sub>.

### 3.3 HRTEM Study

The microstructural information of the samples was obtained using transmission electron microscopy (TEM). Figure 5a shows a TEM image of ZnTiO<sub>3</sub> grown on Si (a, c) (A) and



**Fig. 4** Reflectivity of ZnTiO<sub>3</sub> nanostructures with different thickness grown on PSi substrate

PS (b) (B). As can be seen, the ZnTiO<sub>3</sub> was fully crystalline at the nanoscale and the formation of irregular spherical shaped. The TEM images (Fig. 5) suggest that the submicron-sized particles of A and B are both crystalline with no apparent defects and dislocations. The average diameters of the smallest visible isolated particle/crystallite agglomerate were found to range between 100 nm and 120 nm for A and the particles B with the size of about 80 nm. These results are in excellent agreement with the experimental of values obtained from XRD.

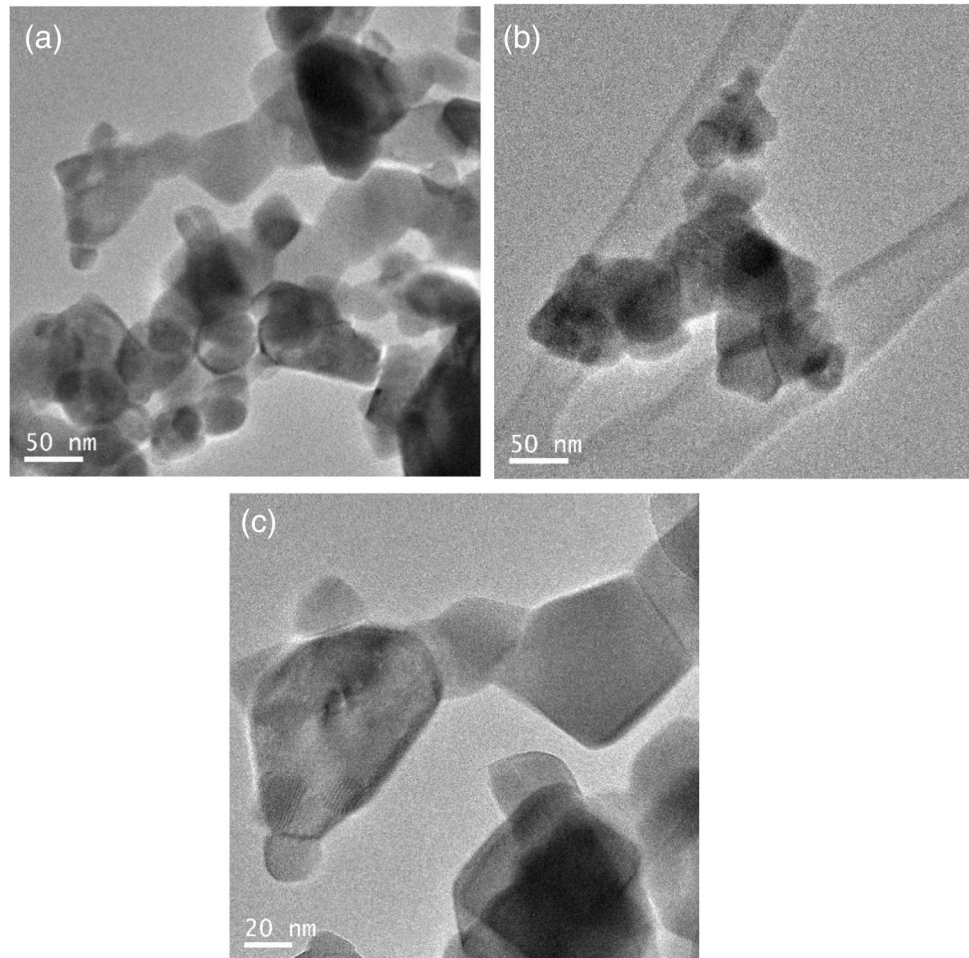
Figure 6 shows surface morphology of ZnTiO<sub>3</sub> particle. Note that the microstructure of B becomes a sponge. This last composed of nanopores. On the other hand, the structure is smooth and lacks pores for A. These results are in parallel with UV-Vis results. The porous structure of ZnTiO<sub>3</sub> influences the increases light absorption (UV-Vis) compared to A (Fig. 2).

The HRTEM image of the sample is presented in Fig. 7. The distance between the adjacent lattice fringes is 0.38 and 0.1 nm for A and B, which can be assigned to the interplanar distance of the hexagonal phases of A and B. Compared with compound A, the crystallinity of B is better. Comprehensively, the results confirmed that the ZnTiO<sub>3</sub> layers were successfully prepared at annealing temperatures at 700 °C.

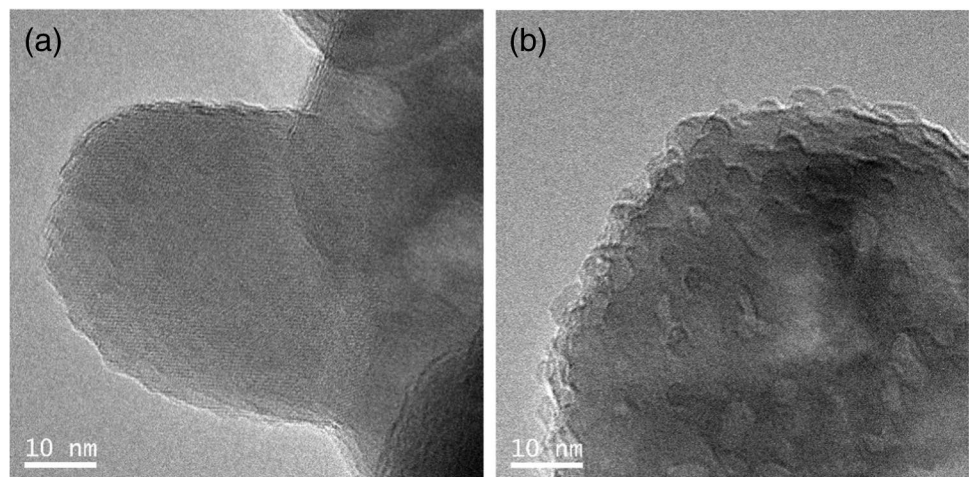
## 4 Conclusion

In summary, we have synthesized nanoscale ZnTiO<sub>3</sub> on PS and Si. The dependence of the structural and optical properties of these nanostructures on the different substrates was investigated systematically. The

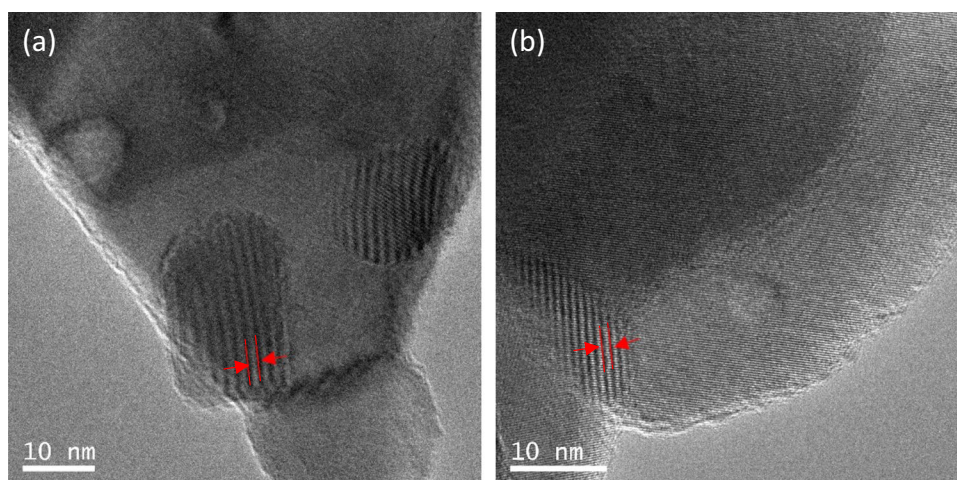
**Fig. 5** TEM image of the  $\text{ZnTiO}_3$  grown on Si (a, c) and PS (b)



**Fig. 6** TEM image of the surface  $\text{ZnTiO}_3$  grown on Si (a) and PS (b)



**Fig. 7** HRTEM images of ZnTiO<sub>3</sub> grown on Si (a) and PS (b)



nanostructures were polycrystalline in nature, (410) plane was the preferred orientation, and showed decreasing crystal grain size with porous silicon substrates. A strongest UV-Vis absorption intensity for ZnTiO<sub>3</sub> nanoscale have been obtained on PS substrate compared to ZnTiO<sub>3</sub> as grown on Si substrate. This is due to the capillary effect and its high specific surface area of PS. With HRTEM, We compared the structure and morphology of ZnTiO<sub>3</sub> nanostructures grown on porous silicon and silicon substrate. The average diameter and the distance between the adjacent lattice fringes of these ZnTiO<sub>3</sub> on porous silicon decreased from 120 to 80 nm and from 0.38 to 0.1 nm, respectively.

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**Data Availability** Not applicable.

## Declarations

**Ethics Approval** Not applicable.

**Consent to Participate** Not applicable.

**Consent for Publication** The Author hereby consents to the publication of the work in the “Silicon” journal.

**Competing Interests** The author declares that there is no conflict of interest in the printing of this manuscript.

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