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(Received 29 June 2025; revised 16 November 2025; accepted 21 November 2025)

Comprehensive Structural and Stress Analysis of ZnO/SiC/porous-Si/Si Multilayer Heterostructures Synthesized Via Sequential Deposition Techniques

Abstract. This research explores the fabrication and structural characteristics of ZnO/SiC/porous-Si/Si multilayer heterostructures synthesized through a controlled multi-step deposition process. The study combines electrochemical porosification of monocrystalline Si substrates, solid-phase epitaxial growth of silicon carbide films, and magnetron sputtering of ZnO layers under varied oxygen partial pressures. Two samples of ZnO films were synthesized under distinct oxygen atmospheres: 0.06 Pa and 0.1 Pa. Comparative XRD analysis reveals that films deposited at lower pressure (0.06 Pa) exhibit enhanced crystallinity, indicated by reduced peak broadening and distinct polycrystalline features. Residual stress analysis confirms compressive biaxial stress in both samples (-0.511 GPa and -0.287 GPa), indicating high crystalline quality and structural integrity of the ZnO films. These findings highlight the effectiveness of buffer layering and deposition control for optimizing ZnO film properties on complex silicon-based architectures.

Keywords: ZnO thin film, porous silicon, silicon carbide, heterostructure, residual stress, structural characterization, X-ray diffraction.

Introduction

Zinc oxide (ZnO) is a II–VI semiconductor with a wide band gap (~ 3.3 eV) and high exciton binding energy (~ 60 meV), which makes it effective in photonics, sensors, and optoelectronics. Thin films and nanostructures of ZnO are effectively utilized as luminescent materials, transparent electrodes, sensitive layers in gas and biosensors, catalysts, and detectors of ultraviolet, X-ray, and gamma radiation.

However, the morphology, structure, and performance characteristics of ZnO depend significantly on the conditions and technology used in its synthesis. In particular, the choice of substrate plays a critical role, considerably affecting the quality of epitaxial ZnO films and the parameters of the resulting devices.

ZnO-based film materials are actively used in optoelectronics, in particular as transparent electrodes, in sensor devices, phosphors and photodetectors sensitive to UV, X-ray and gamma radiation, as well as

in gas analysis systems [1-3]. The quality of ZnO film depends largely on the substrate parameters and deposition method. Traditionally, sapphire Al_2O_3 and silicon Si substrates are used for ZnO growth [4, 5]. While sapphire ensures high crystalline quality, its dielectric properties limit the efficiency of electronic devices based on ZnO/ Al_2O_3 structures. A viable alternative is the ZnO/Si heterostructure, which is more compatible with microelectronic technology. Despite the promising nature of the ZnO/Si system, the difference in lattice parameters and thermal expansion leads to the formation of mechanical stresses and defects at the interface. Works [6-8] have shown that the characteristics of the ZnO crystal structure depend significantly on the deposition conditions, including temperature, partial pressure of oxygen, source power, and process duration. Optimization of these parameters allows for a reduction of the dislocation density, grain size, and residual stresses in the films. The authors of [9, 10] note that the presence of

a buffer layer provides the most effective reduction of stress in ZnO films.

The pore morphology of porous silicon not only facilitates stress relaxation but also significantly influences the adhesion of subsequent layers, including ZnO. Studies have shown that pore size, distribution, and surface roughness affect nucleation and bonding strength at the ZnO/porous-Si interface, thereby impacting film uniformity and epitaxial quality [11-13].

Porous silicon partially relieves mechanical stress due to its elastic structure, but high-temperature ZnO deposition in oxygen atmosphere causes surface oxidation and hampers epitaxial growth [14, 15]. One promising solution is the use of buffer layers, particularly a layer of silicon carbide (SiC) [16, 17], which provides better resistance to high-temperature processing. Silicon carbide is a wide-gap semiconductor ($E_g \approx 2.3\text{-}3.3$ eV depending on the polymorph) that has high thermal conductivity, chemical and thermal stability, and resistance to aggressive environments [18, 19]. Its thermal expansion coefficient ($\sim 4.0 \times 10^{-6}$ K $^{-1}$) is closer to the value for ZnO, which allows for a significant reduction in thermomechanical stresses in the heterostructure [20].

Despite these advances, the combined influence of porous substrates, SiC buffer layers, and deposition parameters on defect formation, residual stress, and structural properties of ZnO films in ZnO/SiC/porous-Si/Si heterostructures remains insufficiently understood. A deeper understanding of these interdependencies is essential for improving the reliability

of heterojunctions in sensors, optoelectronics, and next-generation energy devices.

Solid-state epitaxy enables the deposition of high-quality SiC films compatible with silicon substrates. This compatibility is especially enhanced when a mesoporous structure is pre-formed on the silicon surface [21-23]. Compressive biaxial stresses, which are often found in such structures, can be reduced by using a porous layer and a SiC buffer layer acting as a damping interface [23, 24].

In this regard, it is of great importance to analyze how the combination of a porous substrate, a SiC buffer layer, and deposition modes affects defect formation, residual stresses, and structural properties of ZnO films in the ZnO/SiC/porous-Si/Si heterostructure. This will allow improving the technologies for creating reliable heterojunctions for sensors, optoelectronics, and new generation energy devices.

This work aims to study the defects arising during the formation of zinc oxide heterostructures on SiC/Si substrates with a pre-deposited system of mesopores.

Materials and Methods

The conditions for obtaining zinc oxide films on silicon substrates with previously deposited layers of silicon carbide and porous silicon are described in detail in [25]. The sequence of technological stages is shown in Fig. 1. As a result, two types of samples were obtained, differing in the conditions of ZnO film deposition, as described in [26].

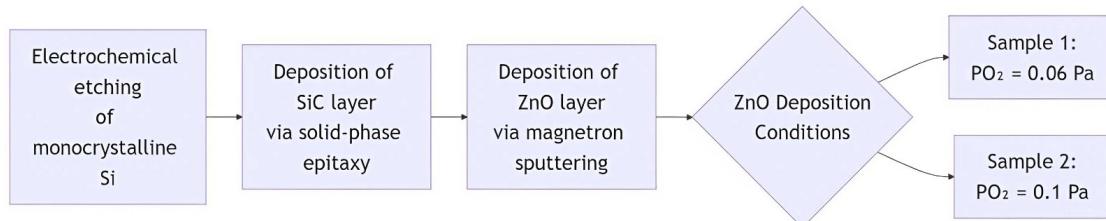


Figure 1 – Sequence of processes for manufacturing ZnO/SiC/porous-Si/Si heterostructure

The article presents the results of the study of samples using scanning electron microscopy (Tescan Mira 3 LMU and Zeiss Supra 25), Raman spectroscopy (Raman) and X-ray diffraction (Philips X'Pert PRO – MRD). The diffraction patterns were obtained in sliding geometry with a scanning

step of 0.03° and an accumulation time of 1.5 s per point. Phase identification was carried out using the ICDD database. The structure of SiC films was studied on an EMR-100 electron microscope in the «reflection» mode at an accelerating voltage of 75 kV.

Results and Discussion

After the process of electrochemical etching, the silicon substrates were completely covered with pores (Fig. 2, a). After annealing in the furnace, silicon with all its numerous pores is covered with a SiC film (Fig. 2, b).

The EDAX energy dispersive X-ray spectrum confirms the presence of elements Si (50.1%) and C (49.3%) and a small amount of O (0.6%) on the sample's surface (Fig. 2, b).

These SEM images confirm the successful formation of a porous silicon layer and its subsequent coverage with a uniform SiC film. The absence of surface defects suggests good compatibility between the porous substrate and the SiC layer.

In the presence of a nanoporous layer, carbon monoxide penetrates to the depth of this layer. At the same time, silicon atoms are replaced by carbon atoms in the columns of nanoporous silicon, and a SiC molecule and a silicon vacancy are formed. The porous layer turns into a SiC film, under which pores form due to silicon vacancies (Fig. 3).

Fig. 3 schematically illustrates this transformation process. It shows how carbon atoms substitute silicon atoms within the porous matrix, leading to the formation of a continuous SiC layer. The model also explains the emergence of subsurface voids beneath the SiC film, which contributes to stress relaxation in the heterostructure.

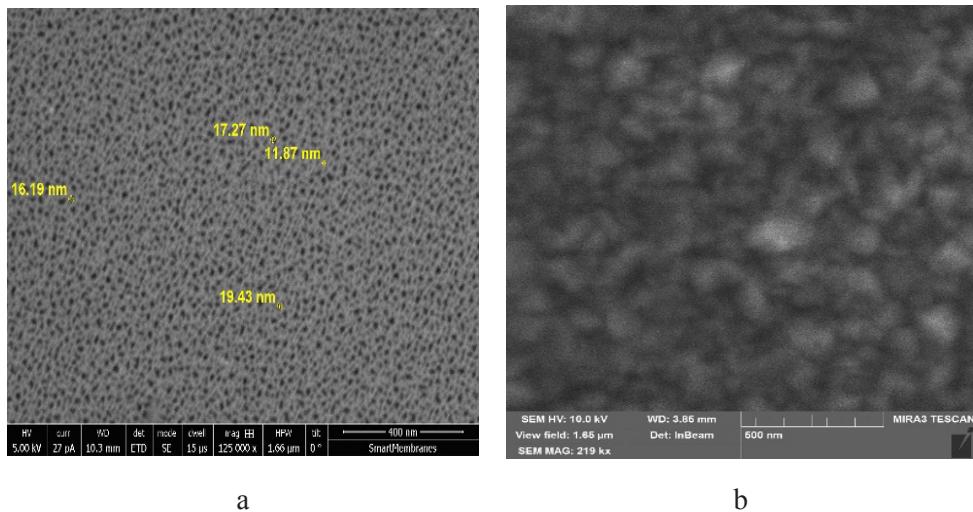


Figure 2 – Surface morphology of porous Si (a) and SiC film (b), observed using SEM

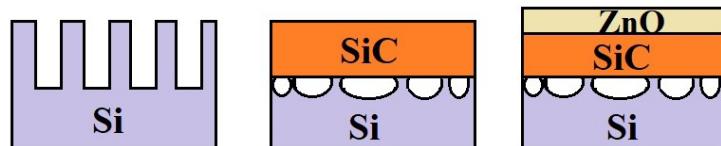


Figure 3 – Model depicting the formation stages of the ZnO/SiC/porous-Si/Si heterostructure

Correct selection of the thickness of the porous-Si layer allows setting the appropriate thickness of the silicon carbide film. At the same time, the pores under the SiC film contribute to reducing mechanical stress in the material.

Fig. 4 displays the electron diffraction pattern from the (100) surface of SiC/Si films along the [110] direction. Spot reflections confirm epitaxial

SiC growth on silicon. The film surface is smooth, without signs of twinning or polycrystalline rings, and the presence of Kikuchi lines confirms its high crystalline quality and epitaxial nature.

The presence of Kikuchi lines and sharp diffraction spots indicates epitaxial growth of SiC on the porous-Si/Si substrate, confirming high crystalline quality.

ZnO layer formed via magnetron sputtering, confirmed by SEM in Fig. 5.

The cross-sectional images reveal uniform ZnO coverage and distinct layer interfaces. Sample 1 (Fig. 1, a) shows a denser and more continuous ZnO layer, consistent with its higher crystallinity and lower stress.

Fig. 6 shows the XRD patterns of ZnO films for sample 2 and sample 1. In sample 2, a diffraction peak is observed at 34.37° with an FWHM of 0.731° , whereas sample 1 exhibits a peak at 34.40° with a narrower FWHM of 0.582° . The presence of a triplet within $31\text{--}36^\circ$ in sample 1 confirms the polycrystalline hexagonal phase of ZnO [6].

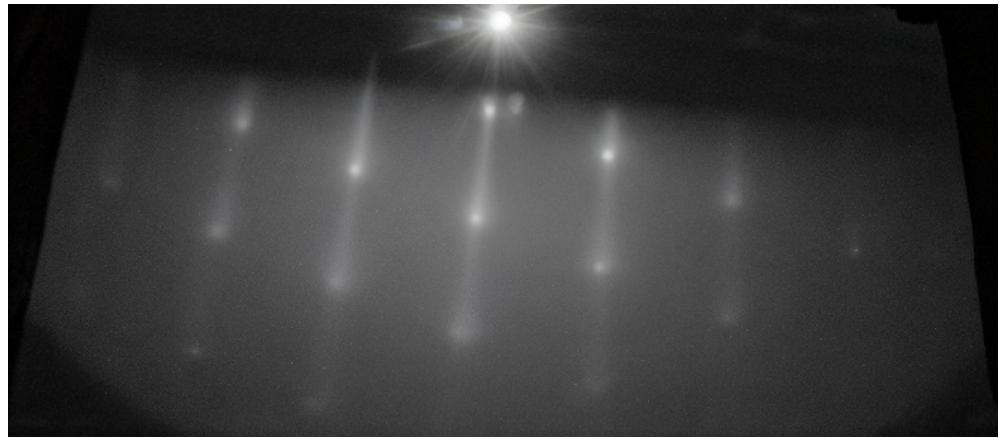


Figure 4 – Electrogram from the surface of the SiC layer

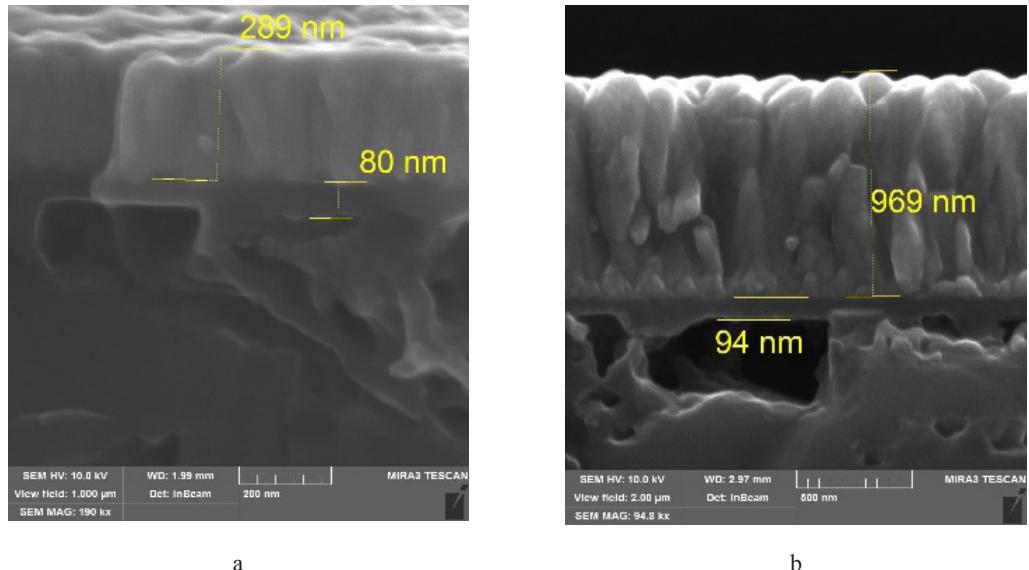


Figure 5 – Cross-sectional SEM images of the ZnO/SiC/porous-Si/Si heterostructure:
(a) sample 1, (b) sample 2

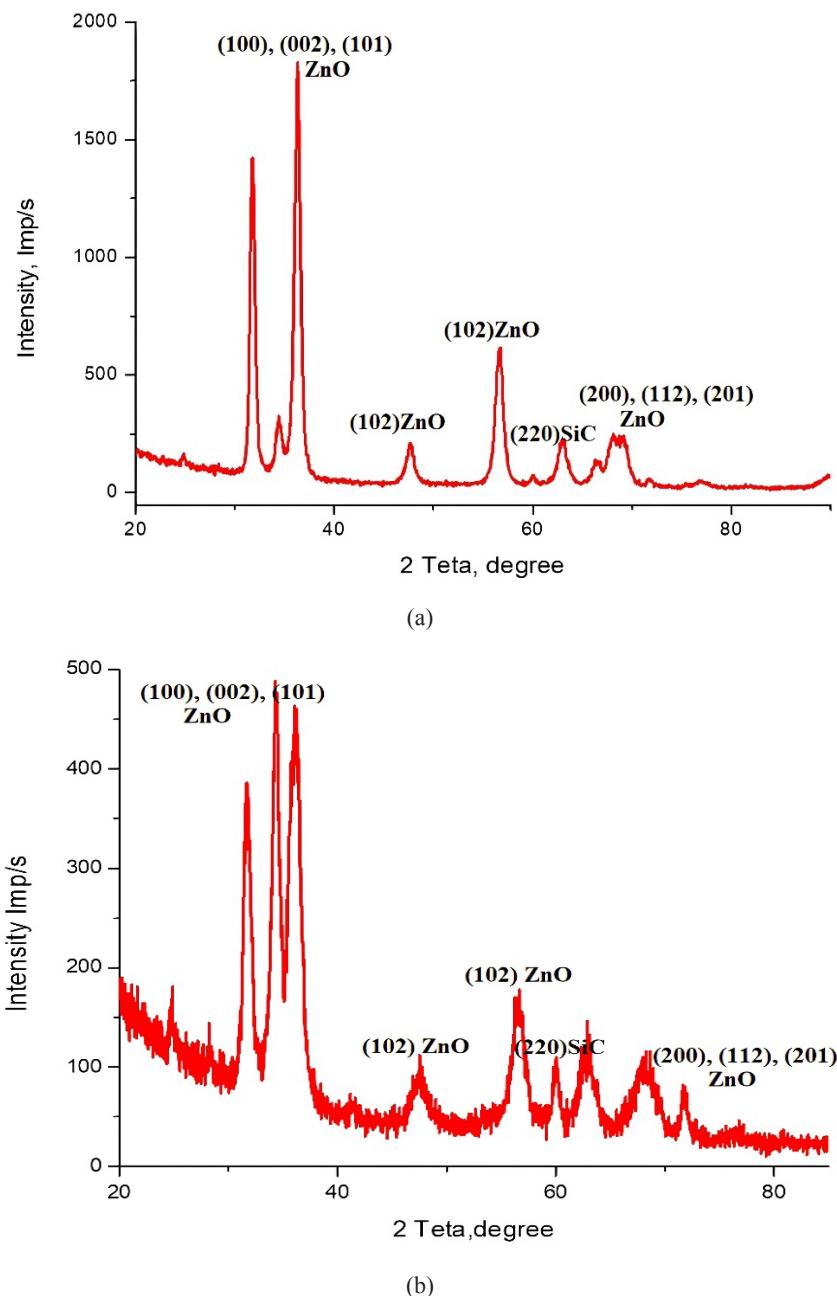


Figure 6 – XRD patterns of ZnO/SiC/porous-Si/Si samples:

(a) sample 1 and (b) sample 2

The triplet observed in sample 1 (Fig. 6,a) confirms the presence of a polycrystalline hexagonal ZnO phase. The shift in peak position and narrowing of FWHM further support improved structural ordering under reduced oxygen pressure.

X-ray diffraction analysis confirms the nanocrystalline structure of ZnO films. The average crystallite size was estimated from the broadening of the

(002) diffraction peak using the Scherrer approach, as described in [7, 26, 27]. The lattice parameter c is calculated from the equation in [28].

Strain and dislocation density were additionally assessed based on peak profile analysis following the methodology reported in [27, 29].

Table 1 shows the results of calculations of the parameters of ZnO films.

Table 1 – Structural parameters of the ZnO films

Parameter	Value	
	Sample 1	Sample 2
β , (°)	0.731	0.582
2Θ (°)	34.37	34.405
a, nm	0.31955	0.31924
c, nm	0.52183	0.52113
d, nm	0.261	0.261
L, nm	0.195	0.195
Grain size, nm (XRD)	11.89	14.94
$\epsilon \times 10^{-3}$	2.63	2.09
$\delta \times 10^{11}$ 1/nm ²	0.0064	0.00403
σ , GPa	-0.511	-0.287

The improved crystallinity of ZnO films deposited at 0.06 Pa can be attributed to enhanced adatom mobility under reduced oxygen partial pressure, which promotes more ordered crystal growth and reduces defect density. This is reflected in the narrower XRD peak and larger crystallite size observed in sample 1. The presence of the SiC buffer layer plays a critical role in stabilizing the ZnO (002) texture by mitigating lattice mismatch and thermal expansion differences between ZnO and the silicon substrate. Furthermore, a clear inverse correlation is observed between crystallite size and residual stress: sample 1, with larger crystallites, exhibits lower compressive stress (-0.287 GPa), suggesting that grain coarsening contributes to stress relaxation in the film.

Negative residual stress values confirm biaxial compressive stress in the structure. The relatively low magnitude of this stress suggests that the obtained ZnO films are of high structural quality. The obtained crystallite size (14.94 nm) and low compressive stress (-0.287 GPa) in sample 1 compare favorably with values reported in earlier studies. For instance, Bouzourâa et al. [16] demonstrated that increasing the porosity of silicon substrates improves ZnO adhesion but does not fully eliminate residual stress, with

grain sizes remaining below 12 nm. The presence of buffer layers contributes to the reduction of residual stresses in the ZnO film compared to Al₂O₃ [5] and Si [30] substrates. Nakamura et al. [13] showed that introducing buffer layers significantly enhances crystallinity and reduces stress in ZnO films grown on silicon, although their work focused on planar Si substrates without porosity. Our results extend these findings by showing that the combination of porous-Si and a SiC buffer layer leads to larger crystallites and lower stress. Moreover, the pronounced (002) texture observed in our films is consistent with the results of Ting et al. [10], who reported that buffer layers promote preferential orientation. Notably, our multilayer architecture achieves these improvements under reduced oxygen pressure, suggesting a synergistic effect of substrate engineering and deposition control.

Conclusion

A comprehensive analysis of ZnO/SiC/porous-Si/Si heterostructures demonstrated the influence of multilayer engineering and deposition parameters on crystallinity and stress levels in ZnO films. The use of porous silicon combined with a SiC buffer layer allowed for a gradual relaxation of lattice mismatch-induced stresses, contributing to uniform film morphology and improved crystalline quality. XRD data confirmed the presence of a stable hexagonal phase, and structural parameters such as dislocation density and strain varied depending on oxygen pressure during ZnO sputtering. The methodology offers a promising route toward fabricating stress-resilient, high-quality ZnO-based heterostructures suitable for sensing and optoelectronic applications.

Acknowledgments

The study was partially supported by the German Research Foundation under the Walter Benjamin Programme, referenced as KI 2865/1-1.

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