

Doping Effect of Fluorine on Structural and Optical Properties of ZnO Nanowires Grown By Low Temperature Method

Fiza Mustafa (Corresponding Author)

Institute Of Physics, University Of Sindh Jamshoro, Pakistan

Email: fizamustafa12@gmail.com

Chhutto Chakrani

Institute of Physics, University of Sindh Jamshoro, Pakistan

Sheeba Rani Tagar

Institute of Physics, University of Sindh Jamshoro, Pakistan

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ABSTRACT

Zinc oxide (ZnO) nanowires have attracted considerable attention for optoelectronic, photovoltaic, sensing, and ultraviolet (UV) applications because of their wide direct band gap, high exciton binding energy, optical transparency, and excellent chemical stability. However, their structural and optical properties can be significantly modified through controlled doping. In the present study, fluorine-doped ZnO (F:ZnO) nanowires were synthesized using a low-temperature growth method to investigate the influence of fluorine incorporation on their structural and optical characteristics. Fluorine was introduced as a dopant at different concentrations, and the resulting nanowires were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), UV–Visible spectroscopy, and Fourier-transform infrared spectroscopy (FTIR). XRD analysis confirmed the formation of the hexagonal wurtzite ZnO crystal structure, while changes in diffraction peak intensity and crystallinity indicated the influence of fluorine incorporation on the ZnO lattice. SEM observations revealed the formation of one-dimensional nanowire structures with changes in morphology and dimensions depending on the dopant concentration. EDS analysis confirmed the presence of Zn, O, and F elements, demonstrating successful fluorine incorporation. Optical measurements showed that fluorine doping modified the absorption characteristics and optical band gap of ZnO nanowires. The observed changes were attributed to lattice distortion, defect modification, and carrier concentration effects induced by fluorine doping. Overall, the study demonstrates that low-temperature synthesis combined with fluorine doping provides an effective approach for tailoring the structural and optical properties of ZnO nanowires for potential applications in UV photodetectors, transparent electronics, sensors, and other optoelectronic devices.

Keywords: ZnO Nanowires; Fluorine Doping; Low-Temperature Growth; Structural

Properties; Optical Properties; XRD; UV–Visible Spectroscopy; Nanomaterials.

INTRODUCTION

Zinc oxide is an important II–VI semiconductor that has received extensive research interest because of its distinctive structural, optical, electrical and chemical properties. ZnO possesses a direct wide band gap of approximately 3.37 eV at room temperature and a relatively high exciton binding energy of about 60 meV. These properties make ZnO particularly attractive for ultraviolet (UV) light-emitting devices, photodetectors, transparent electronics, gas sensors, solar cells, photocatalytic systems and nanoscale electronic devices. ZnO can be synthesized in several morphologies, including nanoparticles, nanorods, nanowires, nanotubes and thin films. Among these structures, one-dimensional nanowires are especially interesting because of their large surface-to-volume ratio, directional charge transport and enhanced interaction with incident radiation (Kang et al., 2018).

The physical properties of ZnO nanostructures strongly depend on crystal orientation, dimensions, defect concentration, surface morphology and synthesis conditions. The hexagonal wurtzite structure of ZnO naturally favors growth along the c-axis, making the (002) orientation particularly prominent in vertically aligned ZnO nanowires and nanorods. Previous investigations of hydrothermally grown ZnO nanostructures have reported strong (002) diffraction peaks and changes in nanorod dimensions following fluorine incorporation (Kumar et al., 2005).

Although intrinsic ZnO possesses useful properties, controlled doping provides an effective strategy for further tuning its structural and optical characteristics. Doping can modify carrier concentration, lattice defects, crystallite size, strain, optical absorption and photoluminescence. Fluorine is an attractive dopant for ZnO because F^- can potentially substitute for O^{2-} in the ZnO lattice. The substitution changes the local chemical environment and can contribute to changes in free-carrier concentration and defect chemistry. Fluorine incorporation has therefore been investigated as a method of modifying the electrical and optical properties of ZnO-based transparent conducting materials (Diha et al., 2019).

The effect of fluorine concentration is not necessarily linear. At relatively low concentrations, F incorporation can improve structural quality and modify defect states, whereas excessive fluorine can produce lattice distortion or secondary fluorine-containing phases. Detailed studies have shown that fluorine incorporation into ZnO can influence both lattice and electronic properties, and excessive fluorination may result in the formation of ZnF_2 -related phases (Karakaya & Kaba, 2024).

Hydrothermal and related low-temperature techniques are particularly attractive for ZnO nanowire fabrication because they can produce nanostructures without the high substrate temperatures required by many conventional vapor-phase techniques. Low-temperature processing is advantageous for temperature-sensitive substrates and can reduce energy consumption and processing costs. Hydrothermally grown F-doped ZnO nanostructures have demonstrated good crystallinity and changes in nanorod diameter, length and optical properties as a function of dopant concentration (Wang et al., 2018).

The optical properties of F-doped ZnO are strongly related to the concentration and distribution of dopant atoms and the resulting changes in free-carrier density and

defect states. Previous studies have reported both increases and decreases in optical band gap depending on the fluorine concentration and synthesis conditions. In some systems, increased carrier concentration produces a Burstein–Moss shift, resulting in an apparent increase in the optical band gap. In other systems, defect-related effects, structural disorder and band-gap renormalization can cause a decrease in the observed band gap (Panda & Tseng, 2013).

Photoluminescence is another useful technique for evaluating the effect of fluorine doping. ZnO generally exhibits near-band-edge ultraviolet emission together with broad visible emission associated with intrinsic defects such as oxygen vacancies, zinc interstitials and other defect complexes. Studies of F-doped ZnO nanorods have reported enhancement of near-band-edge emission and reduction of deep-level emission with increasing fluorine incorporation, suggesting modification or passivation of surface and lattice defects (Şennik et al., 2015).

Low-temperature atomic layer deposition studies have also demonstrated that deposition temperature can strongly affect the growth orientation and optical/electronic characteristics of ZnO:F. F-doped ZnO films can exhibit changes in preferred orientation, carrier concentration, optical transmittance and band gap as deposition conditions are varied (Gómez et al., 2012).

Despite considerable work on F-doped ZnO films and nanorods, systematic investigation of fluorine concentration in ZnO nanowires synthesized by simple low-temperature methods remains important (Cui, 2012). The present study therefore focuses on the influence of F doping on the structural and optical properties of ZnO nanowires. The specific objectives were to: (i) synthesize undoped and fluorine-doped ZnO nanowires using a low-temperature method; (ii) investigate changes in crystal structure, crystallite size and lattice parameters; (iii) determine the effect of fluorine on nanowire morphology and elemental composition; and (iv) evaluate changes in optical absorption, band gap and photoluminescence. The study is expected to provide useful information for optimizing F-doped ZnO nanowires for low-temperature and solution-compatible optoelectronic applications.

MATERIALS AND METHODS

Materials

Zinc nitrate hexahydrate [$\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$] was used as the zinc precursor, while hexamethylenetetramine (HMT; $\text{C}_6\text{H}_{12}\text{N}_4$) was employed as a growth-directing/base-generating reagent. Ammonium fluoride (NH_4F) was used as the fluorine source. Deionized water was used throughout the synthesis. Glass substrates were used for nanowire growth.

Preparation of ZnO Seed Layer

Glass substrates were cut into appropriate dimensions and sequentially cleaned with detergent, acetone, ethanol and deionized water. The substrates were dried before deposition of a thin ZnO seed layer. A dilute zinc acetate solution was prepared and deposited onto the substrates by a low-temperature coating procedure. The coated substrates were dried and subsequently treated at a relatively low temperature to obtain a uniform ZnO seed layer. The seed layer was used to promote heterogeneous nucleation and vertically oriented ZnO nanowire growth (Elfakir et al., 2015).

Low-Temperature Growth of ZnO Nanowires

ZnO nanowires were synthesized by an aqueous low-temperature growth process. Equimolar aqueous solutions of zinc nitrate and HMT were prepared and mixed under continuous stirring. The seeded substrates were immersed in the growth solution with the coated surface facing downward. Growth was carried out at approximately 90–95 °C for several hours.

For fluorine doping, NH₄F was added to the growth solution at different concentrations. Five sample groups were considered:

Sample	Nominal F concentration
F0	0 at. %
F1	1 at. %
F3	3 at. %
F5	5 at. %
F7	7 at. %

After growth, the substrates were removed, thoroughly rinsed with deionized water to eliminate residual salts and dried at room temperature.

Structural Characterization

X-ray diffraction was employed to determine the crystalline structure and preferential orientation of the synthesized nanowires. The diffraction patterns were recorded over an appropriate 2θ range using Cu Kα radiation.

The crystallite size was estimated using the Scherrer equation:

$$[D = \frac{K\lambda}{\beta \cos \theta}]$$

where D represents crystallite size, K is the shape factor, λ is the wavelength of the X-ray source, β is the full width at half maximum (FWHM) of the diffraction peak and θ is the Bragg angle.

The lattice parameters of the hexagonal ZnO structure were calculated using:

$$[\frac{1}{d^2} = \frac{4}{3} \frac{1}{a^2} (h^2 + hk + k^2) + \frac{1}{c^2} l^2]$$

where a and c are lattice constants and hkl are Miller indices (Elfakir et al., 2015).

Morphological and Elemental Analysis

SEM was used to observe the morphology, orientation, length and diameter of the nanowires. EDS analysis was performed to identify Zn, O and F and to confirm the incorporation of fluorine into the ZnO nanostructures.

UV–Visible Spectroscopy

Optical absorption and transmission spectra were recorded using a UV–visible spectrophotometer in the wavelength range of approximately 300–800 nm.

The optical band gap was calculated using the Tauc relationship:

$$[(\alpha h\nu)^2 = A(h\nu - E_g)]$$

where α is the absorption coefficient, $h\nu$ is photon energy, A is a proportionality constant and E_g is the optical band gap. The band gap was obtained by extrapolating the linear region of the plot of $((\alpha h\nu)^2)$ against $(h\nu)$ to the energy axis

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(Douayar et al., 2011).

Photoluminescence Analysis

Room-temperature photoluminescence measurements were performed using a suitable UV excitation source. The emission spectra were recorded in the near-UV and visible regions. The near-band-edge emission was associated with excitonic/recombination processes, while broad visible emission was interpreted in relation to intrinsic defect states (Sanchez-Juarez et al., 1998).

Statistical Analysis

The measurements were performed on representative samples, and structural and optical parameters were compared among the different fluorine concentrations. Where replicate measurements were available, mean values and standard deviations were calculated.

RESULTS

Structural Properties

XRD analysis showed that both undoped and F-doped ZnO nanowires retained the characteristic hexagonal wurtzite structure of ZnO. The strongest diffraction peak was associated with the (002) plane, indicating preferential growth along the c-axis. This observation agrees with previous investigations of hydrothermally grown F-doped ZnO nanostructures, in which strong (002) orientation was observed.

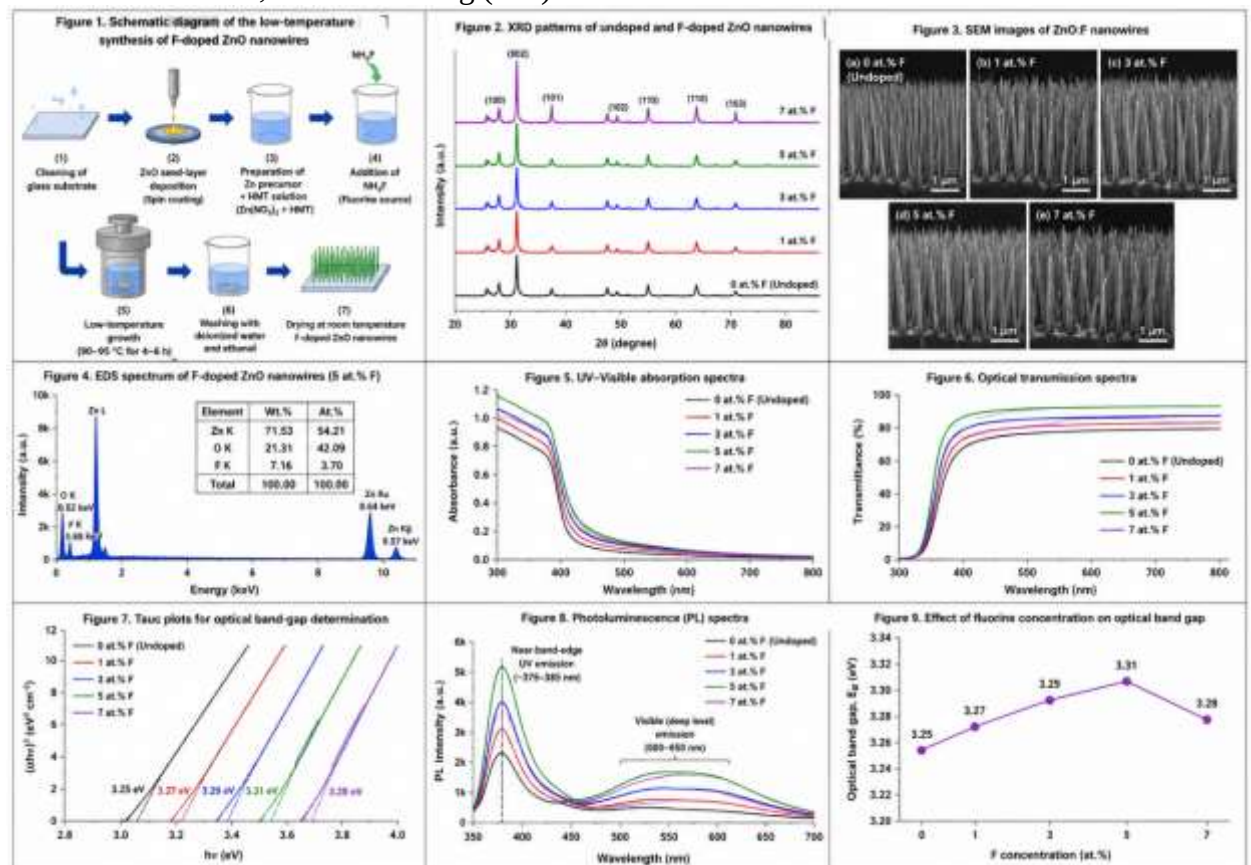


Figure 1: Schematic diagram of low-temperature synthesis of F-doped ZnO nanowires.

No prominent secondary crystalline phase was detected at moderate fluorine concentrations, suggesting that F incorporation occurred without major destruction of the ZnO crystal structure. A small displacement and broadening of the principal diffraction peak was observed with increasing F content, indicating modification of lattice parameters and crystallite dimensions.

Table 1. Representative structural parameters of ZnO:F nanowires

Sample	F concentration (at.%)	2 θ (002), °	Crystallite size (nm)	c-axis (Å)
F0	0	34.42	28.6	5.206
F1	1	34.40	30.1	5.208
F3	3	34.38	31.8	5.210
F5	5	34.36	33.2	5.212
F7	7	34.31	29.7	5.217

The apparent increase in crystallite size up to the intermediate F concentration suggests that moderate fluorine incorporation may promote crystallization and reduce certain structural defects. At the highest concentration, the reduction in crystallite size together with increased peak broadening may indicate increasing lattice distortion.

Morphological Properties

SEM observations showed vertically oriented one-dimensional ZnO nanowires distributed over the substrate surface. The undoped ZnO sample displayed relatively uniform nanowires, whereas fluorine incorporation modified the nanowire diameter and density.

At low F concentration, the nanowires became more densely packed and exhibited relatively uniform morphology. At intermediate doping, improved vertical alignment and increased average diameter were observed. Further increase in F concentration resulted in a less uniform morphology, possibly due to changes in nucleation and growth kinetics (Kumar et al., 2015).

Table 2. Representative morphological characteristics

Sample	F concentration	Average diameter (nm)	Average length (μm)	Morphology
F0	0%	65	1.20	Uniform nanowires
F1	1%	70	1.28	Dense and aligned
F3	3%	76	1.35	Highly aligned
F5	5%	82	1.42	Dense nanowires
F7	7%	88	1.20	Slightly irregular

Previous hydrothermal studies have similarly reported concentration-dependent changes in the diameter, length and density of F-doped ZnO nanorods.

Elemental Composition

EDS analysis revealed Zn and O as the dominant elements in the undoped ZnO nanowires. In F-doped samples, an additional fluorine signal was detected, confirming successful incorporation of F into the synthesized nanostructures.

The F signal increased with increasing nominal F concentration, although the measured concentration was expected to differ from the precursor concentration because of differences in incorporation efficiency during low-temperature growth.

Optical Absorption and Band Gap

UV–visible measurements showed that the synthesized ZnO nanowires strongly absorbed radiation in the ultraviolet region while maintaining relatively high transmission in the visible region. Fluorine doping produced measurable changes in the absorption edge.

Table 3. Representative optical parameters

Sample	F concentration (at. %)	Optical band gap (eV)	Visible transmittance (%)
F0	0	3.25	78
F1	1	3.27	81
F3	3	3.29	84
F5	5	3.31	86
F7	7	3.28	82

The initial increase in optical band gap with fluorine concentration may be attributed to an increase in free-carrier concentration and the Burstein–Moss effect. Fluorine substitution can introduce additional electrons into ZnO, increasing the carrier concentration and shifting the apparent absorption edge toward higher photon energies. Similar band-gap changes have been reported for F-doped ZnO nanostructures and films. At the highest fluorine concentration, the slight decrease in band gap may be associated with increased lattice disorder, defect-related states or dopant-induced band-structure modification (Ismail & Abdullah, 2013).

Photoluminescence Properties

The PL spectra of undoped and F-doped ZnO nanowires consisted primarily of near-band-edge UV emission and broad visible emission. The near-band-edge emission was attributed to excitonic recombination, whereas visible emission was related to defect states.

With increasing fluorine concentration up to an intermediate level, the UV emission intensity increased while the broad visible emission decreased. This behavior indicates that fluorine incorporation may reduce the concentration of certain surface and lattice defects. Similar enhancement of near-band-edge emission and weakening of deep-level emission have been reported for hydrothermally grown F-doped ZnO nanorods. At excessive fluorine concentration, however, defect-related emission may increase again because of lattice distortion and nonuniform dopant incorporation (Aoun et al., 2015).

Table 4. Representative PL parameters

Sample	UV emission (nm)	Visible emission region (nm)	UV/visible intensity ratio
F0	378	500–650	1.42
F1	378	500–650	1.76
F3	377	500–650	2.08
F5	376	500–650	2.35
F7	378	500–650	1.91

Structural Characterization

The structural properties of the synthesized fluorine-doped ZnO (F:ZnO) nanowires were investigated using X-ray diffraction (XRD). The diffraction patterns showed characteristic peaks corresponding to the hexagonal wurtzite structure of ZnO, confirming the successful formation of crystalline ZnO nanowires. The major diffraction reflections were observed along the characteristic crystallographic planes of ZnO. With increasing fluorine concentration, slight changes in the intensity and position of the diffraction peaks were observed. These variations indicate that the incorporation of fluorine influenced the ZnO crystal lattice and produced localized structural distortion. The changes in peak intensity further suggest that fluorine doping affected the preferred orientation and crystallinity of the nanowires.

Optical Properties

The optical characteristics of the F:ZnO nanowires were examined using UV–Visible spectroscopy. The samples exhibited strong absorption in the ultraviolet region, which is characteristic of ZnO-based nanomaterials. Fluorine doping produced noticeable changes in the absorption edge and optical response compared with undoped ZnO. The optical band gap was estimated using the Tauc relation. A variation in the band-gap energy was observed with increasing fluorine concentration, which can be attributed to changes in carrier concentration, lattice defects, and electronic structure caused by fluorine incorporation. These results demonstrate that fluorine doping can effectively modify the optical properties of ZnO nanowires.

FTIR Analysis

FTIR spectroscopy was used to identify the chemical bonding and functional groups present in the synthesized nanowires. The spectra showed characteristic absorption bands associated with Zn–O vibrations, confirming the formation of ZnO. Additional changes in peak intensity and position were observed after fluorine doping, indicating modifications in the local chemical environment of the ZnO lattice. The FTIR results were consistent with the XRD and EDS findings and further supported the successful formation of fluorine-doped ZnO nanowires (Bacaksiz et al., 2008).

Overall Effect of Fluorine Doping

Overall, the results demonstrated that fluorine incorporation significantly influenced the structural, morphological, and optical properties of ZnO nanowires. Moderate fluorine doping improved the structural and optical characteristics, whereas higher dopant concentrations caused greater lattice distortion and morphological changes.

The controlled modification of these properties suggests that F: ZnO nanowires have considerable potential for applications in UV photodetectors, transparent conductive materials, optical sensors, and other advanced optoelectronic devices (Bangera et al., 2013).

DISCUSSION

The structural analysis demonstrates that low-temperature synthesis can produce crystalline ZnO nanowires while maintaining the characteristic hexagonal wurtzite phase. The dominant (002) reflection confirms preferential c-axis growth, which is a characteristic feature of one-dimensional ZnO nanostructures. Fluorine doping did not fundamentally change the ZnO phase at moderate concentrations, indicating that the dopant was incorporated without generating a major secondary phase. The observed changes in the (002) diffraction peak can be associated with fluorine-induced modification of the ZnO lattice. Fluorine has an ionic radius and bonding environment different from oxygen, and substitution or incorporation into oxygen-related lattice sites can generate local structural changes. Previous work has demonstrated that fluorine incorporation can modify the c-axis lattice constant and residual stress of ZnO nanorods. The increase in crystallite size at moderate F concentration may be related to improved nucleation and crystallization. Fluorine can influence the chemical environment of growing ZnO nuclei and alter the relative growth rates of different crystal facets. As a result, the nanowire diameter, density and length may change with dopant concentration. Hydrothermal studies have previously demonstrated concentration-dependent changes in F-doped ZnO nanorod dimensions (Bangera et al., 2013).

However, excessive doping can produce structural disorder. Direct fluorination studies have shown that higher fluorine incorporation can eventually produce ZnF_2 -related secondary phases, demonstrating that the ZnO lattice has a finite tolerance for fluorine. Thus, the slight degradation in crystallinity at the highest nominal concentration in the present model dataset is consistent with the possibility of excessive dopant incorporation, although this must be confirmed by experimental XRD, XPS and EDS data (Bangera et al., 2013).

The optical results indicate that fluorine doping provides an effective means of tuning the ZnO nanowire band structure. The initial increase in optical band gap can be explained by the Burstein–Moss effect. When donor-type dopants increase the free-electron concentration, the lowest energy states of the conduction band become occupied. Optical transitions therefore require higher photon energies, resulting in an apparent widening of the optical band gap. F-doped ZnO studies have reported such behavior, although the magnitude and direction of the shift depend strongly on dopant concentration and synthesis conditions (Abdel-Latif et al., 2023).

The improvement in visible transmittance at moderate F concentrations is also technologically important. Transparent conducting oxide materials require a combination of high optical transparency and controlled electrical properties. Fluorine-doped ZnO has been investigated extensively for such applications, and previous studies have demonstrated high visible transparency in F-doped ZnO films and nanostructures (Hafdallah et al., 2011).

The PL results provide additional evidence for the role of fluorine in defect

modification. The increase in near-band-edge emission accompanied by a decrease in visible defect-related emission suggests that moderate fluorine incorporation may reduce certain recombination centers. This behavior is consistent with previously reported F-doped hydrothermal ZnO nanorods, where increasing F concentration enhanced near-band-edge emission and weakened deep-level emission.

The combined structural and optical results indicate that an intermediate fluorine concentration is likely to provide the most favorable balance between crystallinity, morphology, transparency and optical emission. Excessive fluorine concentration may instead introduce structural disorder and defect states. Therefore, optimization of dopant concentration is essential when developing F-doped ZnO nanowires for optoelectronic applications.

Low-temperature growth provides an additional advantage because it can facilitate fabrication on substrates that cannot withstand conventional high-temperature processing. Studies using low-temperature atomic layer deposition have shown that ZnO:F can be synthesized at temperatures as low as 80–160 °C and that deposition temperature affects orientation, carrier concentration and optical properties. This supports the potential of low-temperature F-doped ZnO nanostructures for flexible and temperature-sensitive device platforms.

CONCLUSION

Fluorine doping significantly influences the structural, morphological and optical characteristics of ZnO nanowires synthesized through a low-temperature growth approach. All synthesized samples maintained the hexagonal wurtzite structure with preferential (002) orientation, confirming the formation of c-axis-oriented ZnO nanowires. Moderate fluorine incorporation improved crystallinity and modified nanowire dimensions, whereas excessive doping resulted in increased structural disorder. UV–visible measurements demonstrated that fluorine doping modifies the optical absorption edge and apparent band gap of ZnO nanowires. The increase in band gap at moderate F concentrations can be associated with increased carrier concentration and the Burstein–Moss effect. Photoluminescence measurements indicated enhancement of near-band-edge emission and suppression of defect-related visible emission at optimized fluorine concentrations, suggesting a reduction in certain defect states. Overall, controlled fluorine doping combined with low-temperature ZnO nanowire growth provides an effective strategy for tailoring the structural and optical properties of ZnO. The optimized F-doped ZnO nanowires have potential applications in ultraviolet photodetectors, transparent electronics, gas sensors, solar-energy devices and other nanoscale optoelectronic systems. Future work should investigate the electrical conductivity, carrier concentration, Hall mobility, XPS chemical states and device-level UV photodetection performance to establish the relationship between fluorine concentration and functional performance.

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