

UV irradiation-promoted wet etching of ZnO nanorods to nanotubes

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In this work, UV irradiation was introduced to promote the etching process of ZnO nanorods towards nanotubes. The ZnO nanorods with different diameters were grown on Au cylindrical spirals via hydrothermal decomposition, and then in the presence of UV irradiation, they were chemically etched to nanotubes. The results indicate that the ZnO nanorods with the larger diameter are easily etched to nanotubes due to enough oxygen vacancies (a kind of defect) within the centre parts, but the ZnO nanorods with the smaller diameter are difficult to form tubular structures because of the insufficient oxygen vacancies. With the introduction of UV irradiation, more oxygen vacancies are produced in the ZnO nanorods and hence, the nanorods with the larger diameter are etched more quickly, and the nanorods with the smaller diameter are etched more easily. Additionally, the UV light with the shorter wavelength is more beneficial for the formation of the ZnO nanotubes than the one with the longer wavelength. The results benefit the wet etching of ZnO nanorods towards nanotubes, especially those with smaller diameters.

1. Introduction: Owing to the hollow structure and large surface area, ZnO nanotubes have been extensively applied in enzymatic biosensors [1], gas sensors [2], and dye-sensitised solar cells [3] to increase the adsorption amounts of enzymes, gases, and dyes, and further to improve the performance of the relevant nanodevices. Nowadays, several routes have been put forward to prepare ZnO nanotubes, such as wet chemical etching [4], chemical vapour deposition [5], template-based growth [6], aqueous chemical growth [7], and atomic layer deposition [8]. Wet chemical etching commonly includes the synthesis of ZnO nanorods and subsequent selective dissolution of their centre parts in aqueous alkali, and is more frequently used due to the simple implementation and low cost [9]. However, not all the ZnO nanorods can be chemically etched to nanotubes despite the concentration of the etching solution and the etching time is increased [10]. To be specific, the ZnO nanorods with the diameter >250 nm can be easily etched to nanotubes because sufficient defects are present in their centre parts. However, the ZnO nanorods with the diameter <250 nm are difficult to form nanotubes owing to the insufficient defects [10, 11]. Up to now, there has been no literature reporting an effective solution to the chemical etching of the ZnO nanorods with a smaller diameter.

As is known, the formation of ZnO nanotubes is mainly dependent on the number of defects in the centre parts of the corresponding ZnO nanorods [12]. Therefore, it is very important to raise the number of defects in the ZnO nanorods. Recently, several publications have reported that the oxygen vacancy as a type of defect can be produced when the ZnO nanorods are stimulated under UV irradiation [13, 14]. Accordingly, it is inferred that if UV irradiation is introduced to the chemical etching of the ZnO nanorods, the number of the oxygen vacancies in the ZnO nanorods will be increased and hence, the ZnO nanorods with the larger diameter perhaps are etched more quickly and those with the smaller diameter are etched more easily. To our best knowledge, there is yet no investigation available on boosting the etching process of ZnO nanorods via UV irradiation.

Herein, ZnO nanorods with different diameters were hydrothermally synthesised on the Au cylindrical spirals, and then in the presence of UV irradiation, they were chemically etched in NaOH solution. The effects of the diameter of the ZnO nanorods, UV irradiation time, and the wavelength of the UV light on the formation of the ZnO nanotubes were systematically investigated.

2. Experimental details

2.1. Materials and reagents: The optical-fibre core with a diameter of 125 μm was purchased from Quanzhou Anpon Company. The Au fibre with the diameter of 30 μm was got from Beijing Doublink Solders Co., Ltd. Acetone, absolute ethyl alcohol, zinc nitrate hexahydrate (purity 99.9%), zinc acetate dehydrate (purity 99.9%), hexamethylenetetramine (purity 99.0%), and sodium hydrate (purity 98.0%) were bought from Tianjin Kemiou Chemical Reagent Co., Ltd. Deionised water (resistance $\geq 18.2 \text{ M}\Omega\text{-cm}$ at room temperature) was produced by Europtronic ultrapure water manufacturing system.

2.2. Hydrothermal synthesis of ZnO nanorods: The Au cylindrical spiral (AuCS) was formed with the Au fibre spiralled compactly around the optical-fibre core, referring to our previous work [15]. The ZnO seed solution with a concentration of 0.5 mM was prepared with zinc acetate dehydrate and sodium hydrate dissolved in absolute ethyl alcohol. The AuCS was immersed in the ZnO seed solution for 1 min to deposit ZnO seed, followed by being dried in the air and annealed at 120°C for 10 min. Zinc nitrate hexahydrate and hexamethylenetetramine were mixed in deionised water. Subsequently, the solution was stirred at 1000 rpm and heated to 90°C and hence, the ZnO growth solution was prepared. The ZnO seed-coated AuCS was kept in the ZnO growth solution at 90°C for 2.5 h. After the reaction, the AuCS with the ZnO nanorods (ZnO/AuCS) was taken out from the ZnO growth solution and rinsed with deionised water. To investigate the effect of the diameter of the ZnO nanorods on the formation of the nanotubes, the concentration of the ZnO growth solution was chosen as 50 mM for the nanorods with the smaller diameter and 90 mM for the nanorods with the larger diameter.

2.3. Chemical etching of the ZnO nanorods towards nanotubes: At room temperature (typically 25°C), the ZnO/AuCSs were immersed in 125 mM NaOH solution to dissolve the centre parts of the ZnO nanorods. The etching time is 1.5, 3, 6, and 12 h. Meanwhile, the ZnO/AuCSs were irradiated by an area source of UV light (wavelength 254 nm or 365 nm, power 24 W) during the entire etching process, and the temperature of NaOH solution was always close to the room temperature after UV irradiation was introduced. Subsequently, the ZnO/AuCSs were taken out

from the NaOH solution, followed by being rinsed with deionised water and dried in the air.

2.4. Characterisation of the ZnO nanorods and nanotubes: The morphologies of the ZnO nanorods and nanotubes were characterised with a field emission scanning electron microscope (FESEM, SU8010, Hitachi, Japan). Owing to the bright lateral walls and dark bottom surfaces of the ZnO nanotubes in the FESEM images, the etching of the ZnO nanotubes was qualitatively determined according to the grayscale of the FESEM images. The photoluminescence spectra of the ZnO nanorods and nanotubes were analysed with a time-resolved fluorescence spectrometer (FLS9, Edinburgh). To verify whether UV irradiation induces the generation of the oxygen vacancies or not, the ZnO nanorods were irradiated by the UV light with the wavelength of 254 nm for 1.5, 3, 6, and 12 h, respectively. Subsequently, the ratios of oxygen (O) to zinc (Zn) were determined by an energy dispersion spectroscopy (EDS, SD-402, Apollo, America).

3. Results and discussion

3.1. Characterisation of the ZnO nanorods and nanotubes: Fig. 1 shows the morphologies of the AuCSs, ZnO nanorods, and ZnO nanotubes. Figs. 1a and b display that the well-aligned ZnO nanorods produce a uniform and dense film on the AuCSs, and the nanorods stand perpendicularly on the surfaces of the AuCSs. The left images of Figs. 1c and d indicate that the ZnO nanorods are typical hexagonal structures. The average diameters of these ZnO nanorods are 159 ± 11 nm and 349 ± 21 nm, and the average lengths are 1.64 ± 0.12 μm and 2.66 ± 0.14 μm , respectively. This indicates that the diameters and lengths of the ZnO nanorods enlarge when the concentration of the ZnO growth solution increases. The right images of Figs. 1c and d indicate that in the absence of UV irradiation, the ZnO nanorods with the smaller diameter are nearly not etched. By contrast, the centre parts of the ZnO nanorods with the larger diameter obviously dissolve in NaOH solution and hence, remarkable tubular structures are formed. This demonstrates that the ZnO nanorods with the larger diameter are more readily etched to nanotubes than the nanorods with the smaller diameter. This is mainly because the ZnO nanorods with the larger diameter contain more oxygen vacancies than the nanorods with the smaller diameter, referring to Fig. 2a.

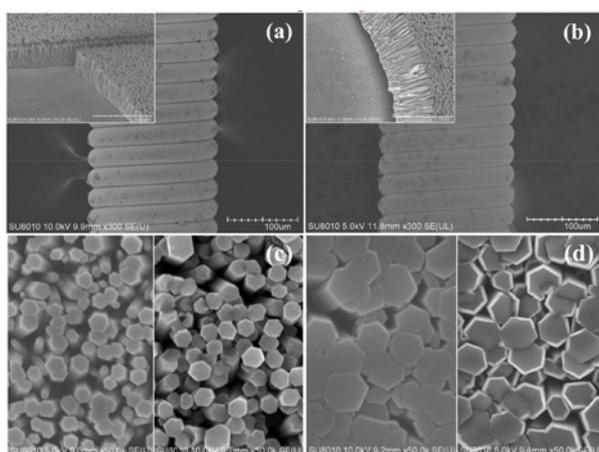


Fig. 1 Morphologies of the ZnO/AuCSs, the ZnO nanorods and nanotubes. a and b FESEM micrographs of the ZnO/AuCSs. The upper left insets are the cross-sections of the ZnO nanorods grown on the AuCSs c and d FESEM micrographs of the ZnO nanorods synthesised in 50 and 90 mM ZnO growth solution, and the ZnO nanotubes etched from ZnO nanorods in 125 mM NaOH solution at 25°C for 3 h. The left images correspond to the ZnO nanorods, and the right images correspond to the related ZnO nanotubes

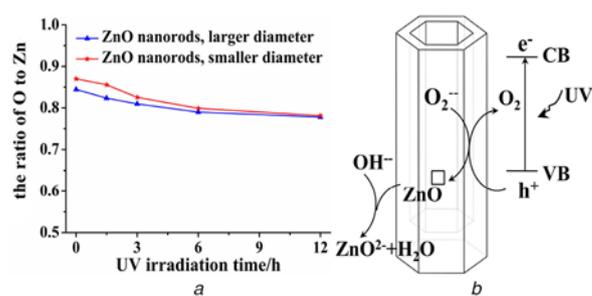


Fig. 2 Effect of UV irradiation on the oxygen vacancy and chemical etching of the ZnO nanorods. a Ratio of O to Zn in the ZnO nanorods that were irradiated by the UV light with the wavelength of 254 nm for 0, 1.5, 3, 6, and 12 h, respectively b Mechanism of the chemical etching of the ZnO nanorods towards nanotubes under UV irradiation

3.2. Effect of UV irradiation on the formation of ZnO nanotubes: The UV irradiation time and the wavelength of the UV light are considered the main factors that influence the formation of the ZnO nanotubes. Since the ZnO nanorods are irradiated by the UV light during the entire etching process, UV irradiation time is equal to the etching time. To unify the terminology, the etching time substitutes the UV irradiation time in the following. The bandgap of ZnO is 3.37 eV at room temperature and hence, the photons with energy at least 3.37 eV (namely the wavelength of the corresponding light is <368 nm) can induce the ZnO nanorods to generate electron-hole pairs [16]. Therefore, UV light with wavelengths of 254 and 365 nm was selected to stimulate the ZnO nanorods.

3.2.1. UV irradiation time: Figs. 3 and 4 show the chemical etching of the ZnO nanorods with the larger diameter that was synthesised in 90 mM ZnO growth solution. Fig. 3 indicates that in the absence of UV irradiation, the ZnO nanorods were gradually etched as the etching time increased. For the etching time 12 h, most ZnO nanorods turned to be perfect ZnO nanotubes, and the lateral walls of several ZnO nanotubes slightly dissolved in NaOH solution. Compared with Fig. 3, Fig. 4 indicates that after UV irradiation was introduced, it only took about 6 h to form perfect ZnO nanotubes, which is 6 h faster than the condition without UV irradiation. Moreover, for the etching time 12 h, most lateral walls

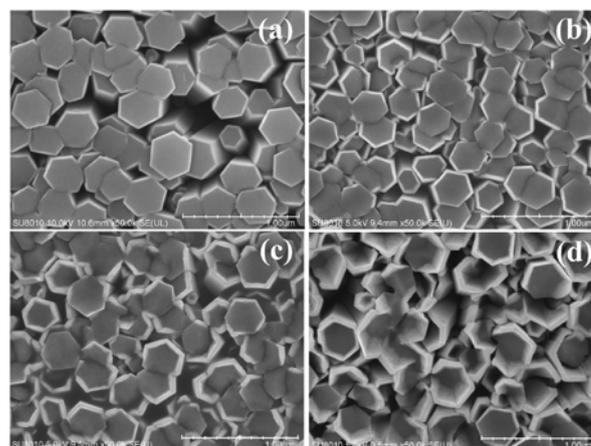


Fig. 3 Chemical etching of the ZnO nanorods synthesised in 90 mM ZnO growth solution in the absence of UV irradiation. The ZnO nanorods were etched in 125 mM NaOH solution at 25°C for a 1.5 h b 3 h c 6 h d 12 h

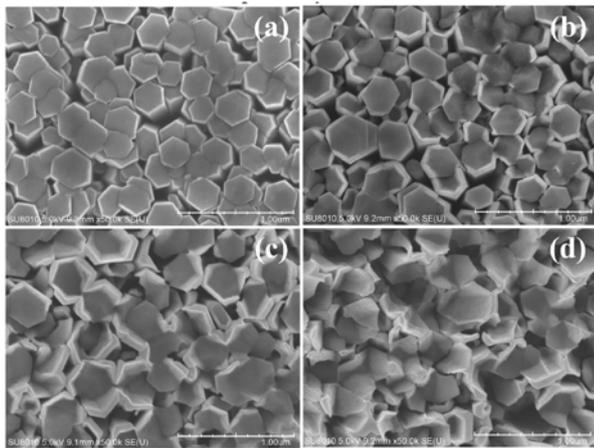


Fig. 4 Chemical etching of the ZnO nanorods synthesised in 90 mM ZnO growth solution in the presence of UV irradiation. The ZnO nanorods were etched in 125 mM NaOH solution at 25°C for
 a 1.5 h
 b 3 h
 c 6 h
 d 12 h. During the entire etching process, the ZnO nanorods were stimulated by the UV light with the wavelength of 254 nm

of the as-formed ZnO nanotubes dissolved in NaOH solution, i.e. the ZnO nanorods were over etched and the as-formed nanotubes were seriously destroyed. These indicate that the etching rate of the ZnO nanorods in the presence of UV irradiation is faster than that in the absence of UV irradiation. Therefore, it demonstrates that UV irradiation accelerates the etching process of the ZnO nanorods with the larger diameter and promotes the formation of the related ZnO nanotubes.

Figs. 5 and 6 show the chemical etching of the ZnO nanorods with the smaller diameter that were synthesised in 50 mM ZnO growth solution. Fig. 5 indicates that in the absence of UV irradiation, even though the ZnO nanorods were immersed in NaOH solution for 12 h, their centre parts only slightly dissolved. However, after UV irradiation was introduced, the ZnO nanorods were obviously etched with the increase of the etching time, referring to Fig. 6. To be specific, the centre parts of the ZnO nanorods started to dissolve in NaOH solution for the etching

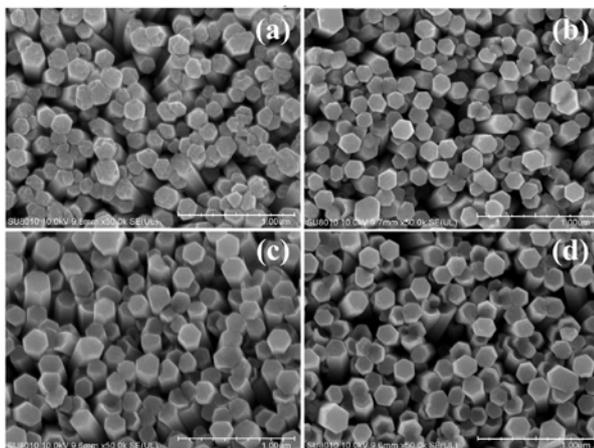


Fig. 5 Chemical etching of the ZnO nanorods synthesised in 50 mM ZnO growth solution in the absence of UV irradiation. The ZnO nanorods were etched in 125 mM NaOH solution at 25°C for
 a 1.5 h
 b 3 h
 c 6 h
 d 12 h

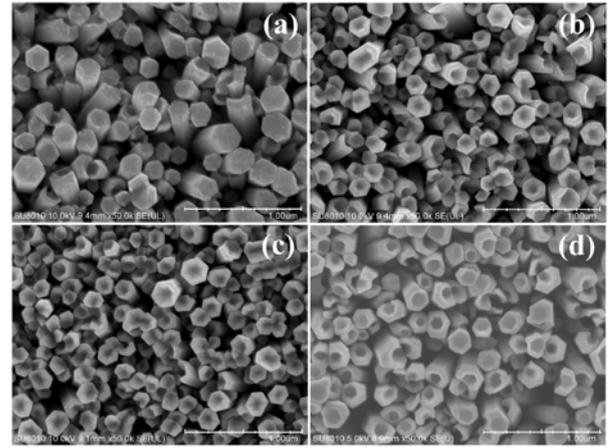


Fig. 6 Chemical etching of the ZnO nanorods synthesised in 50 mM ZnO growth solution in the presence of UV irradiation. The ZnO nanorods were etched in 125 mM NaOH solution at 25°C for
 a 1.5 h
 b 3 h
 c 6 h
 d 12 h. During the entire etching process, the ZnO nanorods were stimulated by the UV light with the wavelength of 254 nm

time 1.5 h. Further, obvious tubular structures appeared for the etching time 3 and 6 h, and perfect ZnO nanotubes were produced for the etching time 12 h. This indicates that UV irradiation also facilitates the etching process of the ZnO nanorods with the smaller diameter remarkably and also enables these nanorods to form nanotubes easily.

3.2.2. Wavelength of the UV light: Comparing with Figs. 3 and 4, 7 exhibits that the ZnO nanorods with the larger diameter were also etched to typical nanotubes for the etching time 6 h. Further, for the etching time 12 h, although most lateral walls of the as-formed ZnO nanotubes dissolved in NaOH solution, several complete ZnO nanotubes were still observed. This suggests that the UV light with the wavelength of 365 nm also accelerates the chemical etching of the ZnO nanorods with the larger diameter, but it promotes the

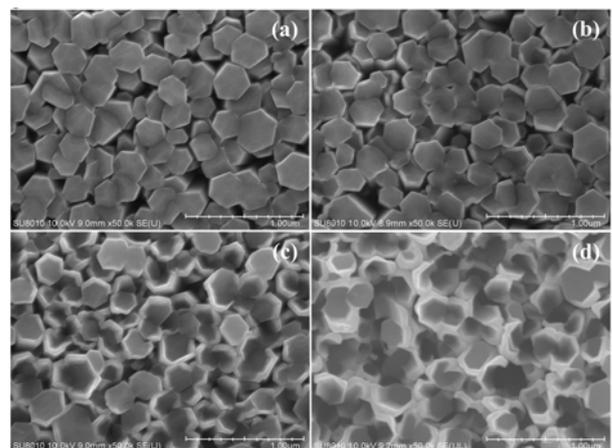


Fig. 7 Effect of the wavelength of the UV light on the chemical etching of the ZnO nanorods synthesised in 90 mM ZnO growth solution. The ZnO nanorods were etched in 125 mM NaOH solution at 25°C for
 a 1.5 h
 b 3 h
 c 6 h
 d 12 h and meanwhile were irradiated by the UV light with the wavelength of 365 nm

centre parts to dissolve in NaOH solution more slowly than that with the wavelength of 254 nm. This is mainly because the UV light with the wavelength of 365 nm possesses lower energy than that with the wavelength of 254 nm; thus the former UV light produces fewer oxygen vacancies than the latter one.

Similarly, in comparison to Figs. 5 and 6, 8 indicates that the centre parts of the ZnO nanorods with the smaller diameter also began to dissolve in NaOH solution for the etching time 1.5 h, and tubular structures were gradually formed as the etching time increased. However, the depths of the tubular structures in Figs. 8b–d were much smaller than those in Figs. 6b–d, thus the as-formed tubular structures in Figs. 8b–d were not as obvious as those in Figs. 6b–d. Therefore, it also concludes that UV light with the shorter wavelength accelerates more quickly the chemical etching of the ZnO nanorods with a smaller diameter than that with the longer wavelength.

3.3. Photoluminescence spectra of the ZnO nanorods and nanotubes: Figs. 9a–c and 10a–c display the photoluminescence spectra for the ZnO nanorods and nanotubes acquired at room temperature. Obviously, all the photoluminescence spectra contained a UV emission peak at 380 nm, and two visible emission peaks at 497 and 633 nm. The UV emission at 380 nm was aroused from the intrinsic recombination of the electron–hole pairs within ZnO [17], and the visible emissions at 497 and 633 nm were caused by the defects in ZnO, such as the oxygen vacancies and interstitial zinc [18, 19]. When the etching time increased, the intensity of the three peaks, especially for that at 633 nm, decreased significantly. This is because the centre parts of the ZnO nanorods dissolved in NaOH solution, which resulted in a reduced amount of the defects in the ZnO nanorods. Since the peak at 633 nm was closely related to the oxygen vacancies [19, 20] and its intensity decayed most remarkably with the increase of the etching time, this peak was selected as the representative to quantitatively investigate the effect of UV irradiation on the chemical etching of the ZnO nanorods, as indicated in Figs. 9d and 10d. The intensity of this peak reduced in the order of $I_{\text{(without UV)}} < I_{\text{(with UV, 365 nm)}} < I_{\text{(with UV, 254 nm)}}$. Therefore, the results once more demonstrate that UV irradiation facilitates the centre parts of the ZnO nanorods to dissolve in NaOH solution, and the UV light with the shorter wavelength is more beneficial for the formation of the ZnO nanotubes.

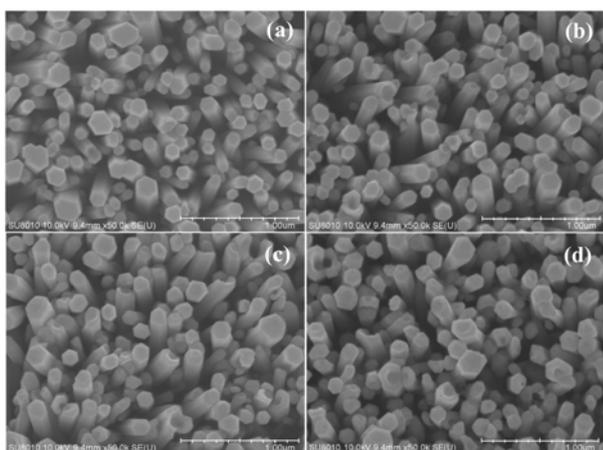


Fig. 8 Effect of the wavelength of the UV light on the chemical etching of the ZnO nanorods synthesised in 50 mM ZnO growth solution. The ZnO nanorods were etched in 125 mM NaOH solution at 25°C for
a 1.5 h
b 3 h
c 6 h
d 12 h and meanwhile were irradiated by the UV light with the wavelength of 365 nm

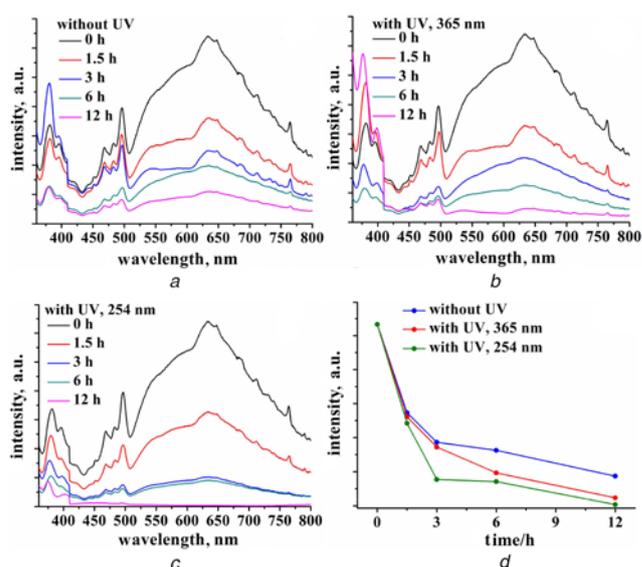


Fig. 9 Effect of UV irradiation on the photoluminescence spectra of the ZnO nanotubes evolved from the nanorods with the larger diameter.
a–c Photoluminescence spectra of the ZnO nanorods and nanotubes without UV irradiation and with UV irradiation. The ZnO nanorods were synthesised in 90 mM ZnO growth solution
d Intensity of the peak at 633 nm for different etching time

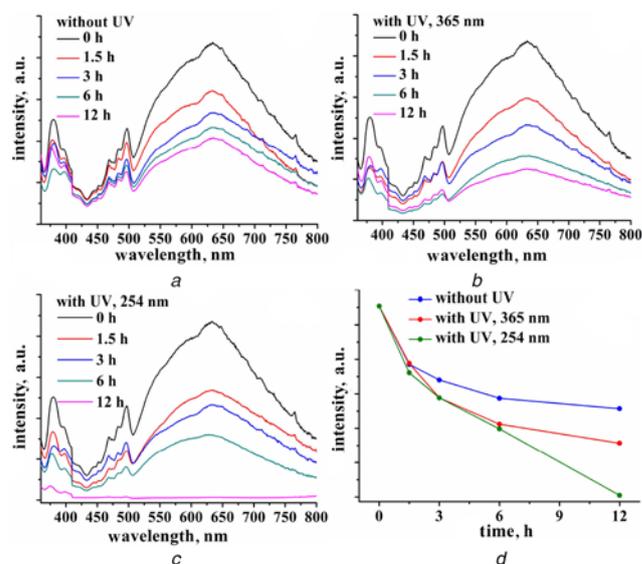
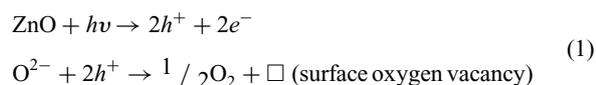


Fig. 10 Effect of UV irradiation on the photoluminescence spectra of the ZnO nanotubes evolved from the nanorods with the smaller diameter.
a–c Photoluminescence spectra of the ZnO nanorods and nanotubes without UV irradiation and with UV irradiation. The ZnO nanorods were synthesised in 50 mM ZnO growth solution
d Intensity of the peak at 633 nm for different etching time

3.4. Mechanism of chemical etching of ZnO nanorods towards nanotubes under UV irradiation: Fig. 2a shows that without UV irradiation, the ratios of O to Zn in the ZnO nanorods with the larger diameter and the nanorods with the smaller diameter are less than one. This suggests that some oxygen vacancies are present in the as-prepared ZnO nanorods. Moreover, the ratio of O to Zn in the ZnO nanorods with the larger diameter is less than that in the nanorods with a smaller diameter. This indicates that more oxygen vacancies appear in the ZnO nanorods with a larger diameter. After the ZnO nanorods were irradiated by the UV light, the ratio of O to Zn decreased. This implies that UV irradiation promotes the generation of oxygen vacancies in

the ZnO nanorods. It is because UV irradiation generates electron-hole pairs in the ZnO nanorods and then these holes (h^+) react with the lattice oxygen (O^{2-}), which gives rise to the formation of surface oxygen vacancies (\square), referring to (1) [13]



With the increase of UV irradiation time, the number of surface oxygen vacancies in the ZnO nanorods raises. This gives rise to more oxygen vacancies in the ZnO nanorods with the larger diameter and sufficient oxygen vacancies in the ZnO nanorod with a smaller diameter. Therefore, the centre parts of the ZnO nanorods with the larger and the smaller diameters become more instable, and dissolve in NaOH solution more quickly than the condition without UV irradiation, as shown in Fig. 2b. As a consequence, the chemical etching of the ZnO nanorods with the larger diameter is accelerated, and that of the nanorods with the smaller diameter starts.

4. Conclusion: In summary, ZnO nanorods with the larger and the smaller diameters were hydrothermally synthesised on Au cylindrical spirals in different concentrations of the ZnO growth solution. Under UV irradiation, these ZnO nanorods were chemically etched in NaOH solution for 1.5, 3, 6, and 12 h, respectively. It turns out that in the absence of UV irradiation, the formation of ZnO nanotubes mainly depends on the diameter of the corresponding nanorods. Moreover, the larger the diameter of the ZnO nanorods is, the more oxygen vacancies exist and further the more easily nanotubes are formed. In the presence of UV irradiation, the oxygen vacancies in the ZnO nanorods are produced and accelerate the chemical etching of the nanorods. Therefore, the ZnO nanorods with the larger diameter dissolve in NaOH solution more quickly, and the nanorods with the smaller diameter are etched to nanotubes more easily. Furthermore, the UV light with the shorter wavelength accelerates the etching process of the ZnO nanorods more remarkable than the one with a longer wavelength. The results provide an effective solution to accelerate the etching process of ZnO nanorods towards nanotubes.

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