液相法制备取向 ZnO 纳米线阵列的场发射特性

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摘要: 采用水热合成工艺, 在不同条件下制备了不同的一维取向 ZnO 纳米线阵列样品. 用 X 射线衍射仪 (XRD)、扫描电镜 (SEM) 及透射电镜 (TEM) 对样品的晶体结构和形貌等进行了表征, 对样品的场发射特性进行了分析和比较, 并用 Fowler-Nordheim 方程对影响 ZnO 纳米线场发射的因素进行了研究. 结果表明, 具有较低生长密度分布、较高的长径比和较尖锐生长端的 ZnO 纳米线阵列样品具有较好的场发射特性.

关键词: ZnO; 纳米线阵列; 场发射; 液相法

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Field Emission Properties of Aligned ZnO Nanowire Arrays Prepared by Simple Solution-Phase Method

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Abstract: One-dimensional (1D) aligned ZnO nanowire arrays with different morphologies were synthesized by a solution-phase method. The morphology and microstructure of the products were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The field emission property of different ZnO nanowire array samples was compared. The factors that influence the field emission property of the 1D ZnO nanowire arrays were analyzed using the Fowler-Nordheim equation. The results showed that the ZnO nanowire samples with the lower areal density, higher aspect ratio, and thin tips showed much better field emission characteristics.

Key Words: ZnO; Nanowire array; Field emission; Solution-phase method

High-quality field emitters are very desirable for applications in a wide range of field-emission-based devices such as flat-panel displays and other electronic devices. 1D ZnO semiconductor nanostructures with its inherent properties of larger length-to-diameter, higher surface-area-to-volume ratio, thermal stability, oxidation resistance, and high chemical stability should be a

good candidate for field emission applications. Recently, the field emission property of different 1D ZnO nanostructures, such as nanowires, nanoneedles, nanopins, and nanotubes has been studied^[1–5]. The previous research on 1D ZnO nanostructure field emitters showed that the shape, aspect ratio, screening effect, and contact behavior (both mechanical and electrical) are the

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primary influence factors on the field emission properties^[6-9]. However, the field emission properties of 1D ZnO nanowires with different geometrical structures still have many obscure problems to deal with. For example, the field emission properties of ZnO nanowire arrays with regular high aspect ratio and the effect of emitter density on the field-screening were rarely studied due to the difficulty in the preparation of those emitters with different densities. Therefore, it is essential to synthesize well-aligned 1D ZnO nanostructures and pursue the physical origins of the dependence of the field emission of 1D ZnO nanowire emitters on the geometrical factors for improving their field emission properties.

There are various methods for synthesizing 1D ZnO nanostructures, such as pulsed laser deposition, thermal evaporation, electrochemical deposition, chemical or physical vapor deposition, solution-phase approach, etc^[10-16]. But, most of the ZnO nanostructures in these published field emission articles were synthesized with the high-temperature synthesizing techniques. The high-temperature techniques, including pulsed laser deposition^[17], chemical vapor deposition (CVD)^[18], and thermal evaporation^[19-20], are energy-consuming and expensive. In most of these studies, the 1D ZnO nanostructures are deposited on the higher resistance silicon and sapphire substrates. Therefore, undesirable, defective contact resistance can be caused between the ZnO nanostructures and substrates. This result is unfavorable to the enhancement in the field emission current density of 1D ZnO nanostructure field emitters.

Recently, the solution-phase approaches to produce high-quality 1D aligned ZnO nanostructures have attracted extensive interest on account of their low growth temperature (<100 °C), low cost, no metal catalyst needed, easy to control, and good potential for scale-up with general substrates^[10-16]. In addition, this solution-phase controlled fabricating approach can grow 1D ZnO nanostructures directly on various metal foils. Then, robust electrical contact can be formed in the growth processes. This better electrical contact is beneficial to rational designs with different sizes for raising the field emission current density of the 1D ZnO nanostructure field emitters. Meanwhile, the controlled fabrication of high-quality ZnO nanostructures with low temperature, facile manipulation, and potential for scale-up can enable the straightforward integration of ZnO nanostructures into nano-electronic devices, such as field emission displays and micro/nanosensors.

In this article, well-aligned 1D ZnO nanowire arrays were fabricated on the zinc foil using a very simple hydrothermal reaction method at a low temperature (95 °C). The comparative investigation on the field emission properties of different ZnO nanowire array samples was carried out. The influence factors of field emission property were analyzed.

1 Experimental

Aligned ZnO nanowire arrays were directly prepared on zinc foils (99.99 %, 0.2 mm thick) reacting in aqueous ammonia solu-

tion. Beforehand zinc foils (10 mm×10 mm) were ultrasonically washed in analytical grade acetone, ethanol, and deionized water for 20 min, successively. The effects of solution concentration and growth time on the microstructure of ZnO nanowire arrays had been studied. (1) Some zinc foils were dipped into corresponding reactive aqueous ammonia for 3 h at room temperature to form a ZnO-seed film, and then the treated zinc foils were vertically immersed into 20 mL aqueous ammonia solution of 4%, 7%, 10%, and 15% (V/V) for 24 h, separately. (2) Some treated zinc foils were reacted in 15% aqueous ammonia for 6, 12, 24, and 48 h. All of the reacting processes were performed in a sealed Teflon reaction kettle (25 mL) heating at a constant temperature of 95 °C.

The obtained ZnO nanowire products were then rinsed with deionized water and dried in air for further characterization. The morphology and microstructure of synthesized nanowires were characterized by X-ray diffraction (XRD, Rigaku DMAX PSPC MDG 2000), scanning electron microscopy (SEM, KYKY-1010), and transmission electron microscopy (TEM, JEOL-2010).

Field emission properties of the different samples were carried out inside a vacuum chamber, which was pumped down to about 3.1×10^{-5} Pa at room temperature. The tests were measured using a simple diode configuration. The cathode was the as-grown ZnO nanowires and the zinc foil was used as a cathode-conducting layer. The anode was polished pure copper rod. The gap between cathode and anode was controlled by the thickness of a mica spacer containing a 2 mm circular hole in the center. Voltages up to 2.5 kV were applied to the anode with a step of 100 V. And the emission current (I) was detected with a micro amperometer. The testing electric field (E) was estimated from dividing the applied voltage (V) by the anode-cathode distance (d). The emission current density (J) was calculated from the obtained emission current and the area of the rounded hole in the mica. The emission current-voltage characteristics were analyzed by using the Fowler-Nordheim equation.

2 Results and discussion

Fig.1(a-d) show low- and high-magnification SEM and TEM images of the ZnO nanowire arrays synthesized in different precursor concentrations of aqueous ammonia solution at 95 °C for 24 h. From the images, large-scale well-oriented ZnO nanowire arrays were observed with uniform and dense arrays. They are approximately of the same length, 8–10 μm. The TEM inset in Fig.1(d) testifies that the ZnO nanowires have a smooth surface without catalytical growth droplets at their growth tips. In each sample, the diameter of ZnO nanowires has little variation from bottom to top. As increase of precursor concentrations from 4% to 15%, the mean diameter of the nanowires altered from 600, 400, 250, to 150 nm, the growth density changed from 2, 6, 9 to 25 μm⁻². And the diameter sizes of the grown ZnO nanowires were reduced and the growth density in a unit area was enhanced. According to these basic values from the SEM images in Fig.1, the specific surface area of the synthesized ZnO

Fig.1 Typical SEM images of ZnO nanowire arrays fabricated in various aqueous ammonia concentrations for 24 h at 95 °C

The insets are low- and high-magnification SEM and TEM pictures of the ZnO arrays and individual nanowires. ammonia concentration: (a) 4%, (b) 7%, (c) 10%, (d) 15%

nanowire arrays in a unit area was calculated. The calculated ratio of specific surface area is about 6.86:30.6:72.9:333. So the specific surface areas of synthesized ZnO samples increased with the increase of precursor concentration from 4% to 15%. Fig.2 shows the corresponding XRD pattern of ZnO nanowire arrays in different aqueous ammonia concentrations for 24 h. The three diffraction peaks of (002), (100), and (101) show good agreement with those of the JCPDS (36-1451) data of the ZnO ($a=0.325$ nm, $c=0.521$ nm). The sharp and narrow (002) diffraction peaks at 34.2° exist in every product. With the increase of aqueous ammonia concentration, the intensity of the (002) diffraction peaks is enhanced. It indicates that the synthesized ZnO materials are highly aligned perpendicular to the substrate with a c -axial growth direction.

The SEM images in Fig.3(a–d) show four different ZnO nanowire arrays prepared at different reacting time. These results represent that growth length of ZnO nanowire arrays are a function of the growth time from 6 to 48 h in 15% aqueous ammonia. As the reaction time was changed from 6 to 48 h, the length-wise growth has experienced from nucleation to short nanowires, to long nanowires. From the initial time to 6 h growth stage, a layer of ZnO growth nucleation with higher distributed density was observed in Fig.3(a). The diameter of the growth nucleation distribution is in the range of 240–300 nm, and the average growth length is about 350 nm. When the reac-

tion time was increased from 12 to 24 h in Fig.3(b–c), ZnO nanowires began to grow along the (002) direction obviously. And the length varied from 3 to 10 μm . When the response time was increased to 48 h, an interesting aspect of the as-grown sample in Fig.3(d) is gained. Some longer and sparse ZnO nanowires with high aspect ratio and thinness of the tips are showed on the top surface of the nanowire arrays. The average length is 40 μm . The inset in Fig.3(d) presents the high magnification image of the samples. We think that the diameter of ZnO nanowires on the growing tips will appear significantly different as the increase of growth time. The result is that a high diversity in

Fig.2 XRD patterns of ZnO nanowire arrays synthesized at 95 °C and 4%, 7%, 10%, and 15% aqueous ammonia for 24 h

Fig.3 SEM images of ZnO nanowire arrays prepared in 15% aqueous ammonia at different growth time
growth time: (a) 6, (b) 12, (c) 24, (d) 48 h; The inset in Fig.3(d) presents the high magnification image of the samples.

growth rate would be present. The nanowires with small-diameter tips have a larger growth rate and the nanowires with large-diameter tips have a lower growth rate. In the latter stage of the ZnO nanowire growth processes, these nanowire arrays appear to form mostly discrete and sparse morphology on the top surface of as-grown ZnO nanowire arrays. When the growth time is long enough, sparse ZnO nanowires with high aspect ratio and thinness of the tips can be represented.

The field emission performances of ZnO nanowire samples

Fig.4 J - E behaviors of ZnO nanowire arrays synthesized in different ammonia solutions for 24 h at 95 °C
ammonia concentration: (a) 4%, (b) 7%, (c) 10%, (d) 15%;
The inset is corresponding Fowler-Nordheim plots of four different ZnO nanowire arrays samples.

synthesized under different conditions have been investigated. Firstly, the effect of diverse ZnO nanowire diameters on their field emission characteristics was studied. Fig.4 gives the J - E characteristic curve of four kinds of ZnO nanowire corresponding to that of Fig.1(a-d). It shows that the field emission current density (J) from the different samples is a function of the applied electrical field (E). For all the samples, as the E is increased, the emission current density J is also elevated, and no saturation of J is evident under the highest E . The turn-on field (E_{ton}) and threshold field (E_{thr}) values of all the samples were evaluated. Generally, E_{ton} and E_{thr} are arbitrarily defined as the electrical fields under which a J of $100 \mu\text{A}\cdot\text{cm}^{-2}$ and $1 \text{mA}\cdot\text{cm}^{-2}$ can be observed, respectively. Table 1 lists the measured values of E_{ton} and E_{thr} for all the samples. It shows that the larger the mean diameter of the ZnO nanowires is, the higher the values of

Table 1 E_{ton} , E_{thr} and β values of ZnO nanowire arrays synthesized in different aqueous ammonia at 95 °C for different time

Sample	Concentration	Growth time (h)	E_{ton} ($\text{V}\cdot\mu\text{m}^{-1}$)	E_{thr} ($\text{V}\cdot\mu\text{m}^{-1}$)	β
1a	4%	24	17	26	1.1×10^3
1b	7%	24	14	24	1.7×10^3
1c	10%	24	13	21	1.9×10^3
1d	15%	24	10	17	2.4×10^3
3a	15%	6	14	22	1.5×10^3
3b	15%	12	11	19	2.0×10^3
3c	15%	24	10	17	2.4×10^3
3d	15%	48	7	11	3.2×10^3

Fig.5 J - E behaviors of ZnO nanowire arrays prepared in 15% aqueous ammonia for different reaction time

reaction time: (a) 6, (b) 12, (c) 24, (d) 48 h.

The inset is corresponding Fowler-Nordheim plots.

their E_{ton} and E_{thr} are attained.

The published article show that electrical contact, emitter geometry and screening effect are three prerequisites for field emission property^[21]. The direct growth of aligned ZnO 1D nanowires on conducting metal substrates *via* a simple solution-phase approach may facilitate their electrical contact with the external circuit. These arrays will, therefore, have smaller E_{ton} and E_{thr} values. Meanwhile, the inset in Fig.4 shows approximate linear relations between $\ln(J/E^2)$ and $1/E$, suggesting that the electron emission could be well formulated by the Fowler-Nordheim theory^[22-23],

$$\ln(J/E^2) = (-B\phi^{3/2}/\beta E) + \ln(A\beta^2/\phi)$$

where, β is the field enhancement factor, ϕ is the work function of the emitter which is 5.3 eV for ZnO material^[24], A and B are constants with the value of $1.56 \times 10^{-10} \text{ A} \cdot \text{V}^{-2} \cdot \text{eV}$ and $6.83 \times 10^3 \text{ V} \cdot \text{eV}^{-3/2} \cdot \mu\text{m}^{-1}$, respectively. The β could be derived from the slope of $\ln(J/E^2) - (1/E)$, and the values of β are estimated and listed in Table 1. It clearly demonstrates that the sample in Fig. 1a has the smallest β value of 1.1×10^3 and the sample in Fig.1d has the largest β value of 2.4×10^3 .

Field emission characteristic is correlated with the morphology of ZnO nanowires. In these field emission experiments in Fig. 4, four ZnO nanowire samples are approximately of the same length, 8–10 μm . We can see from the SEM images that ZnO nanowires in the four samples show high growth density, and the distances between single ZnO nanowire is in the range of 20 nm to tens of nanometer in space. So strong field screening effect will be generated. Then, the worse field emission property with the higher E_{ton} and E_{thr} as well as a relative small β value is produced. These results come from the screening effect of dense growth of 1D ZnO nanowires.

Ku *et al.*^[25] demonstrated that the geometric construction such as tip cone angle (θ) and tip radius (R_{tip}) are two important factors for the field emission property of 1D nanostructure emitter,

i.e., small θ and R_{tip} are beneficial to field emission. Ramgir *et al.*^[6] had reported the theoretical field enhancement factor $\beta_0 = 1/kr$, where k is a constant known as the geometrical factor, and r is the radius. Without doubt, The sample in Fig.1(a) has the largest diameter, so it has the smallest β value. And the sample in Fig.1 (d) has the smallest diameter, so it has the largest β value. On the other hand, the high aspect ratio of the nanowires can generate a high electric field, which decreases the field emission potential barrier and so increases the field emission current^[8]. As a result, ZnO nanowires in Fig.1(d) with smaller diameter and higher aspect ratio have larger β value (2.4×10^3) and thus smaller turn-on field ($10 \text{ V} \cdot \mu\text{m}^{-1}$).

Secondly, the field emission behaviors of ZnO nanowire samples with four different reaction time in 15% aqueous ammonia were measured, which correspond to the SEM images of Fig.3 (a–d). The reaction times are 6, 12, 24 and 48 h and the growth lengths of these ZnO nanowires are about 350 nm, 3, 10 and 40 μm , respectively. Fig.5 gives the J - E curve from these ZnO nanowire samples. The inset is corresponding Fowler-Nordheim plots. Table 1 shows the measured values of E_{ton} , E_{thr} , and β for these samples. It can be seen that the ZnO nanowire arrays fabricated for 6 and 12 h have the worse field emission property with the highest E_{ton} and E_{thr} values, and their β values are about 1.5×10^3 and 2.0×10^3 . Meanwhile, the samples fabricated for 24 h has the better field emission property with the lower E_{ton} and E_{thr} values, and β is about 2.4×10^3 . But the samples fabricated for 48 h has the best field emission property with the lowest E_{ton} and E_{thr} values, and its β value is about 3.2×10^3 . The applied field required to $1 \text{ mA} \cdot \text{cm}^{-2}$ is about $11 \text{ V} \cdot \mu\text{m}^{-1}$. This result is smaller than the corresponding E_{thr} value of $22 \text{ V} \cdot \mu\text{m}^{-1}$ in the samples fabricated for 6 h.

It is well known that the field enhancement factor β reflects the enhanced electron emission due to the localized electronic states and the value of β can be modified by other effects such as the field-screening effect from the proximity of emitters. After obtaining the values of β of the four different samples, we can analyze quantitatively the field-screening effect on the field emission property from the ZnO nanowire arrays with different coverage densities on the top growth surface. In the sample of Fig.3(d), the thinness of the tip, high aspect ratio and lower coverage densities on the top growth surface are appeared. The distance between the adjacent emission sites is enlarged compared with the other samples, which can decrease the electrostatic screening effect and increase the effective emission sites. Therefore, this growth morphology will substantially reduce the result of the screening effect. So the excellent field emission property and the higher β value are gained. But the ZnO nanowire emitters in Fig.3(a, b) are main consisted of a layer of spherical ZnO growth nuclei with high coverage density. And some growth nuclei have not clear growth direction. The local field produced from higher coverage of ZnO emitters will decrease the β value owing to the screening effect^[1]. At the same time, many structur-

al defects and complicated microstructures exist in the nanowire arrays. Thus the higher E_{on} and E_{th} value, the lower J at the same field E value are achieved^[7-9]. So the ZnO nanowire sample in Fig. 3(d) with high aspect ratio, thinness of the tips and sparse microstructure shows better field emission characteristics.

3 Conclusions

In summary, aligned ZnO nanowire arrays were fabricated using a very simple hydrothermal reaction method on conducting zinc metal foils at 95 °C. Better electrical contact property was created between the growth ZnO nanowires and metal substrates. The experimental results show that the field emission characteristics of the ZnO nanowire arrays can be adjusted by the electrical contact and emitter geometry as well as its screening effect. The larger the mean diameter of the ZnO nanowires is, the higher the values of their E_{on} and E_{th} is attained. When the growth time is long enough, sparse ZnO nanowires with high aspect ratio and thinness of the tips can be represented. The field emission screening effect of aligned ZnO nanowire arrays will be reduced. As a result, the as-grown ZnO nanowire arrays synthesized with the hydrothermal reaction method can achieved excellent field emission properties. The above conclusions suggest that these aligned ZnO nanowire arrays have great potential applications in flat panel displays and other electronic devices.

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