

## Controlled cleavage of single semiconducting nanowires and study on the suitability of their use as nanocavities for nanolasers

Q. Chen<sup>a)</sup> and L.-M. Peng

Key Laboratory for the Physics and Chemistry of Nanodevices and Department of Electronics,  
Peking University, Beijing 100871, China

(Received 29 January 2004; accepted 30 March 2004; published online 28 May 2004)

Single semiconducting nanowires have been cleaved to desired length at desired locations inside the scanning electron microscope (SEM) using a nanoprobe system. SEM and transmission electron microscope examinations of the cleaved nanowires revealed that the cleaved ends of the nanowires are in general atomic flat, but not without atomic steps. Possible use of the cleaved nanowire as nanocavity for nanolaser was considered, and several key parameters were estimated. In particular, our result shows that, for a semiconducting CdS nanowire, the effect of the atomic steps at the cleaved ends of the nanowire is negligible if the nanowire cavity is longer than several micrometers. © 2004 American Institute of Physics. [DOI: 10.1063/1.1757635]

Semiconducting nanowires may potentially be used as building blocks in nanoelectronics, optoelectronics, and photonics.<sup>1–7</sup> In particular ultraviolet nanowire nanolasers have been fabricated using ZnO and CdS nanowires.<sup>6,7</sup> Detailed optical and electric measurements on the single-crystal CdS nanowires shown that they can function as Fabry–Perot optical cavities.<sup>7</sup> These optoelectronic applications require, however, that the ends of the nanowires be flat in order to function as reflecting mirrors, and this imposes a severe limitation as to the synthetic methods and types of the nanowires that may be used. Although some nanowires do have flat ends,<sup>6</sup> most of the as grown nanowires do not have flat ends. In principle the method of sonication may be employed to produce flat ends,<sup>7</sup> but this method does not have any control as to where to break the nanowires and on the length of the so formed Fabry–Perot cavities. Here we report an investigation on the controlled cleavage of single nanowires in the scanning electron microscope (SEM), and show that a nanoprobe system may be readily used to produce Fabry–Perot cavity with desired length and therefore offering a possible control on the wavelength of the nanowire nanolaser.

The *in situ* cleavage experiments were carried out using a MM3A nanoprobe system installed in a FEI XL 30F SEM.<sup>8</sup> Figure 1(a) is a photograph showing the MM3A nanoprobe outside the SEM together with a Chinese penny which has a diameter of 1.8 cm. The control system of the nanoprobe allows the tip of the nanoprobe to move on a surface of a sphere with an accuracy of about 2.5 nm, and the radial movement of the tip perpendicular to the surface may be controlled with an accuracy of 0.25 nm. For nanoscale manipulation, sharp tips of nanometer scale are of crucial importance. In this study we used tungsten tips made by etching tungsten wires in NaOH solution, SEM examination of the tips shows that the tips are typically of the size of about 20–50 nm.

Single semiconducting nanowires were found and the movement of the nanoprobe was monitored using the SEM. The tip of the nanoprobe was first positioned at the desired

point on the nanowire with a fine step of movement of about 0.25 nm. The tip was then withdrawn from the nanowire, and a force was exerted on the nanowire by moving forward the tip with a larger step of movement, e.g., 1.0 nm. With suitably positioned tip and added force, the nanowire may be cleaved at the desired position at the nanowire.

The cleaved nanowires were further examined by transmission electron microscope (TEM) (in the present work we used a FEI F30 electron microscope). To enable TEM observations of the same cleaved nanowire we directly dispersed the nanowires onto the holey carbon film on a TEM grid. The grid was then loaded into the SEM, and nanowires were examined by the SEM and cleaved *in situ* by the MM3A nanoprobe system. After the cleavage of the nanowires, the grid sample was moved out of the SEM and transferred into the TEM. The same nanowires were found and observed. Figure 1(b) is a SEM image showing a tip above a ZnO nanowire that was cleaved by the tip inside the SEM.

Semiconducting CdS nanowires used in this study were fabricated using solvothermal method. TEM examination confirmed that the nanowire has a wurtzite structure with  $a = 4.14 \text{ \AA}$  and  $c = 6.72 \text{ \AA}$ .<sup>9</sup> Figure 2(a) is a SEM image showing that most of the CdS nanowires have diameter around 50 nm and most ends of the nanowires are not flat. Figure 2(b) is a TEM image showing a typical nanowire having a curved end. High resolution TEM (HRTEM) image of the nanowire

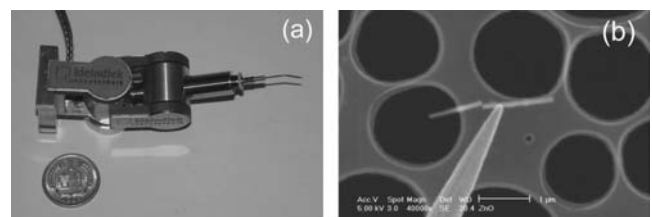


FIG. 1. (a) An external view of a MM3A nanoprobe. (b) SEM image showing the tip of the nanoprobe, together with a cleaved ZnO nanowire.

<sup>a)</sup>Electronic mail: qingchen@pku.edu.cn

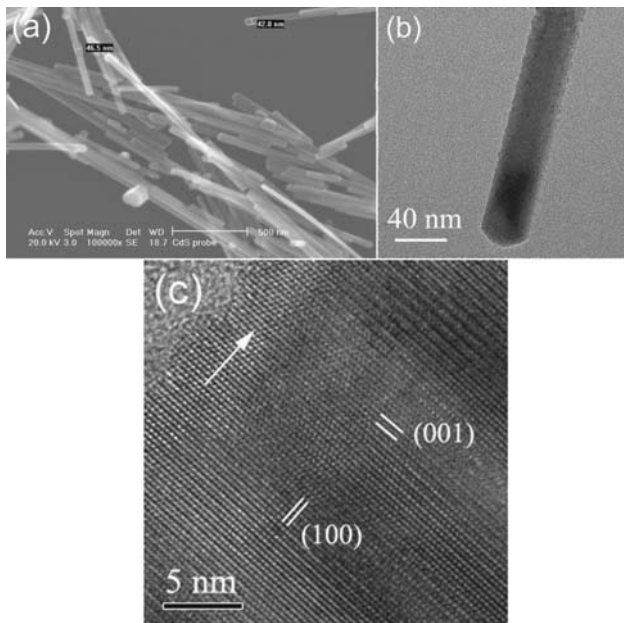


FIG. 2. (a) SEM image showing the CdS nanowires of about 50 nm in diameter. (b) TEM image of one CdS nanowire with round end. (c) HRTEM image showing that the CdS nanowire is single crystal with growing axis (pointed by the arrow) along the [001] direction.

[Fig. 2(c)] shows that the nanowire is single crystal with its axis along the [001] direction. There exist also a few nanowires containing twins [see e.g., the lower nanowire shown in Fig. 3(a)]. But regardless of these twins the axis of the nanowire is along the [001] direction. Figure 3(a) is a TEM image showing two cleaved CdS nanowires. The cleaved ends are seen to be flat and perpendicular to the axes of the nanowires. Shown in Fig. 3(b) is a HRTEM image of the cleaved nanowire shown on top of Fig. 3(a). The cleavage is shown to be on the (002) plane and the cleaved end is atomic flat. However the HRTEM image of Fig. 3(b) shows that the cleaved end is not perfect. An atomic step is also observed at the cleaved end. The height of the step is measured to be 0.67 nm, which is consistent with the spacing of the (001) plane of CdS.

Similar cleavage experiments were also carried out on ZnO nanowires, which were synthesized using physical vapor deposition method. The axis of the nanowires is along [0001]. Most of the as grown ZnO nanowires studied in this work do not have flat ends. Shown in Figs. 4(a) and 4(b) are two SEM images of the same ZnO nanowire taken before

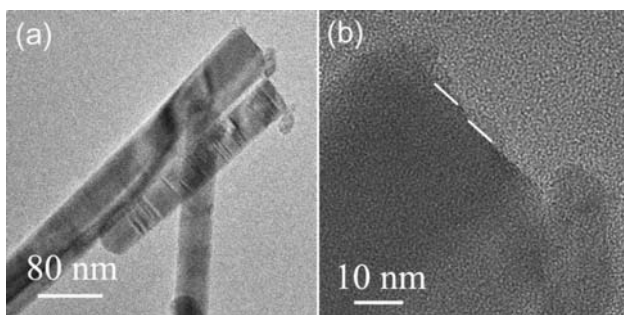


FIG. 3. (a) TEM image of two cleaved CdS nanowires. (b) HRTEM image of the cleaved end of the top nanowire of (a) showing the cleaved surface is atomically flat with one step of 0.67 nm.

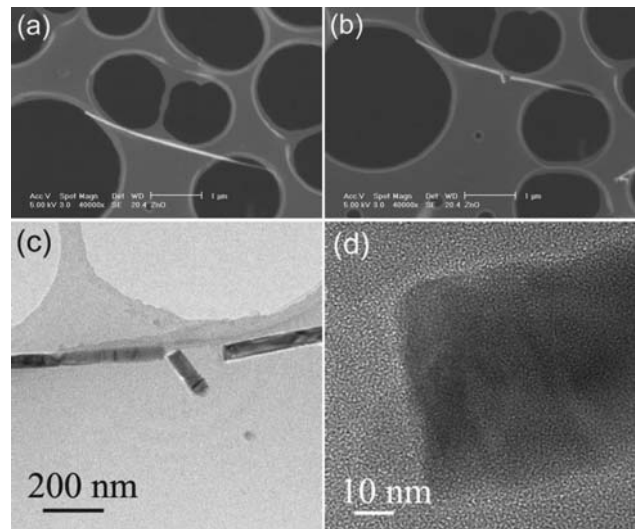


FIG. 4. SEM images taken (a) before and (b) after a ZnO nanowire was cleaved twice by a nanoprobe. (c) TEM image of the same ZnO nanowire as shown in (b), (d) HRTEM image of the right part of the nanowire showing the cleaved end is flat.

and after cleavage. Figure 4(c) is a TEM image showing the same cleaved nanowire, and Fig. 4(d) is a HRTEM image showing the cleaved end of the right part of the nanowire of Fig. 4(c). The cleaved end is seen to be flat, although not without atomic steps.

Cleaved surfaces have been used for a long time as reflection mirrors in resonators in large scale. In some favorable cases the naturally formed faces<sup>6</sup> and cleaved ends via sonication<sup>7</sup> have been proved as being able to act as reflecting mirrors for nanowire resonator. However evidence was not provided as to whether or not steps exist on those flat ends. We now consider the question as to how the presence of a few atomic scale steps would affect the use of the nanowire as lasing resonator, and present some estimations on a number of key parameters using the CdS nanowire as an example. Our estimations show that the effect is negligible if the nanowire cavity is longer than about 2.5 μm.

We considered three cavities with length  $L = 510, 2550,$  and  $15300$  nm. Using the relation  $L = m\lambda / (2n)$ ,<sup>10</sup> where  $L$  is the length of the cavity,  $\lambda$  is the wavelength in the air, and  $n$  is the index of the medium inside cavity, and assuming  $n = 2.5$  and  $\lambda = 510$  nm, we obtain the mode number  $m$  equal to 5, 25, and 150, respectively for the three cavities with different  $L$ . The mode spacing was calculated using  $\Delta\lambda = (\lambda^2/2L)(n - \lambda(dn/d\lambda))^{-1}$ .<sup>11</sup> For a first-order estimation, we have assumed that  $dn/d\lambda = 0$  and obtained  $\Delta\lambda = 102, 20.4,$  and  $3.4$  nm, respectively, for the three  $L$ , indicating that the resonant modes separate further apart for shorter cavities.

The steps at the end of the nanowire may introduce several effects. The length of the cavity will change by the amount of the step height, the reflectance of the end changes when there exist steps at the end, and waves reflected from different parts of the stepped end will have different phases resulting in a mixing of phases in the cavity.

Assuming there exist single atomic steps on both ends, we would then have at least three distances between the two mirrors, i.e.,  $L, L + 0.67$  nm and  $L + 1.34$  nm for a CdS nanowire. The resonance wave with  $\lambda = 510$  nm in the cavity

TABLE I. Estimation of several parameters for Fabry–Perot optical cavities formed by CdS nanowires with different lengths.

Length of cavity (nm)	510	2550	15300
Mode number (for $\lambda = 510$ nm)	5	25	150
Wavelength spacing (nm)	102	20.4	3.4
Wavelength shift due to steps (nm)	1.34	0.27	0.04
FWHM $\delta\lambda$ (nm), without steps	0.66	0.13	0.022
FWHM $\delta\lambda$ (nm), with steps	2.32	0.46	0.077

having  $L = 510$  nm will shift to  $\lambda_s = 511.34$  nm when the two mirrors separated  $L + 1.34$  nm, and  $\lambda_s = 510.27$  and  $510.04$  nm, respectively, for  $L = 2550$  nm and  $L = 15300$  nm. Experimental measurements show that the peak width of stimulated emission from CdS nanowire is around  $0.3$  nm.<sup>7</sup> Therefore, the length effect of the steps can be ignored for cavities longer than  $2.5$   $\mu\text{m}$ .

Imperfect reflection at the mirrors will cause resonance loss and increase the resonance linewidth. The width of the resonance  $\delta\nu$  can be estimated via  $\delta\nu \approx \nu_F/\mathcal{F}$  for  $\mathcal{F} \gg 1$ , where  $\nu_F$  is the spacing between adjacent resonance frequencies,  $\mathcal{F}$  is the finesse of the resonator<sup>10</sup>  $\mathcal{F} = \pi\gamma^{1/2}/(1-\gamma)$ ,  $\gamma^2 = \exp(-2\alpha_r L)$  is the overall intensity attenuation factor,  $\alpha_r = \alpha_s + 1/2L \ln(1/R_1 R_2)$ ,  $\alpha_s$  being the absorption factor, and  $R_1$  and  $R_2$  the reflection coefficients at the two mirrors. Since the steps on the mirrors do not affect the absorption inside the cavity, we may ignore the absorption in the present estimation by assuming  $\alpha_s = 0$  and obtain  $\gamma^2 = R_1 R_2$ . In wavelength space, the full width at half maximum (FWHM) of the resonance peak can be expressed as  $\delta\lambda \approx \Delta\lambda/\mathcal{F}$ , where  $\Delta\lambda$  is the wavelength spacing between resonance modes. Assuming that the two cleaved ends have the same reflectance of  $0.98$  and the step area is  $5\%$  of the total cleaved end area, we have the following results: for perfect cleaved ends with no steps,  $R_1 = R_2 = 0.98$ ,  $\mathcal{F} = 155$ ; for flat ends containing steps,  $R_1 = R_2 = 0.98 \times 95\% = 0.931$ , and  $\mathcal{F} = 44$ . The FWHM of the resonance peak due to the resonance loss may then be calculated and the results are listed in the last two lines of Table I. The increase in the FWHM due to the steps is negligible if the cavity is longer than  $2.5$   $\mu\text{m}$ .

The phase shift caused by the step may be estimated

using  $\phi = 2\pi \times 2\Delta L/(\lambda/n)$ ,  $\Delta L$  being the step height. For  $\Delta L = 0.67$  nm,  $\lambda = 510$  nm, and  $n = 2.5$ , we have  $\phi = 0.0066 \times 2\pi$ , which is much smaller than  $2\pi$ . Therefore the existence of the steps at the surface will not affect the resonance of the waves.

In summary, single semiconducting nanowires have been successfully cleaved at desired points using a nanoprobe system inside the SEM. We estimated the effect of the step on the potential use of the nanowire as nanolaser cavity and found that the effect is negligible as long as the nanowire is longer than several micrometers.

This work was supported by the MOST (Grant No. 001CB610502), NSF of China (Grant Nos. 60271004, 90206021, and 90206048), the Key Project (Grant No. 10401), and EYTP of the Chinese Ministry of Education. We thank M. S. Wang and S. Wang for preparing the tips, Professor Y. Li for providing CdS nanowires, Professor Z. H. Xi for providing ZnO nanowires, and Dr. S. Kleindiek for valuable discussions and technical help with the MM3A nanoprobe system.

<sup>1</sup>J. F. Wang, M. S. Gudiksen, X. F. Duan, Y. Cui, and C. M. Lieber, *Science* **293**, 1455 (2001).

<sup>2</sup>X. F. Duan, Y. Huang, Y. Cui, J. F. Wang, and C. M. Lieber, *Nature* (London) **409**, 66 (2001).

<sup>3</sup>M. Kazes, D. Y. Lewis, Y. Eberstein, T. Mokari, and U. Banin, *Adv. Mater.* (Weinheim, Ger.) **14**, 317 (2002).

<sup>4</sup>H. Kind, H. Yan, B. Messer, M. Law, and P. D. Yang, *Adv. Mater.* (Weinheim, Ger.) **14**, 158 (2002).

<sup>5</sup>M. S. Gudiksen, L. J. Lauhon, J. F. Wang, D. C. Smith, and C. M. Lieber, *Nature* (London) **415**, 617 (2002).

<sup>6</sup>M. H. Huang, S. Mao, H. Feick, H. Yan, Y. Wu, H. Kind, E. Weber, R. Russo, and P. D. Yang, *Science* **292**, 1897 (2001).

<sup>7</sup>X. F. Duan, Y. Huang, R. Agarwal, and C. M. Lieber, *Nature* (London) **421**, 241 (2003).

<sup>8</sup>L.-M. Peng, Q. Chen, X. L. Liang, S. Gao, J. Y. Wang, S. Kleindiek, and S. W. Tai, *Micron* **35**, 495 (2004).

<sup>9</sup>N. A. Razik, *J. Mater. Sci. Lett.* **6**, 1443 (1987).

<sup>10</sup>B. E. A. Saleh and M. C. Teich, *Fundamentals of Photonics* (Wiley, New York, 1991).

<sup>11</sup>K. Iga, in *Handbook of Semiconductor Lasers and Photonic Integrated Circuits*, edited by Y. Suematsu and A. R. Adams (Chapman & Hall, London, 1994), p. 34.