Fabrication of oriented polymeric nanofibers on planar surfaces by electrospinning

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Fabrication of oriented polymeric nanofibers on planar surfaces by electrospinning

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We present a method for the formation of oriented polymeric nanofibers using electrospinning deposition from an integrated microfluidic device. The source includes a microfabricated triangular shaped tip, integrated at the exit of a microfluidic channel to form a source that can be brought close to a counter electrode and scanned over surface features. Using the ability to orient the nanofiber deposition, we formed a 140 nm diameter suspended nanofiber over etched trenches on a silicon wafer, a configuration that allows for electrical and mechanical measurements of deposited nanofibers. © *2003 American Institute of Physics.* [DOI: 10.1063/1.1592638]

Nanoscale materials such as nanowires, nanotubes, and nanofibers have gained more attention recently because of their unique electrical, optical, and mechanical properties.[1–3] Fabrication of oriented nanofibers on a planar surface and integration of these nanofibers with microfabricated structures such as electrodes or surface topography are required for application to molecular electronics.[4–7] Random woven nanofibers with diameters in the range of 50–500 nm have been fabricated using electrospinning technology and suggested for use in a wide rage of applications such as high performance filters,[8,9] drug delivery,[10] scaffolds for tissue engineering,[11] optical and electric applications.[12] Straight nanofibers with diameters ranging from 100 to 300 nm were fabricated by electrospinning on an edge of a sharpened rotational disk collector for possible application to molecular electronics.[13] However, this approach does not permit fabricating of nanofibers on planar surface. Therefore, it is impossible to integrate nanofibers with microfabricated structures for such applications. The method we described here permits the realization of structure and device architectures.

In this letter, we describe an oriented polymeric nanofiber fabrication method by electrospinning a polymeric solution on a moving planar silicon surface and integrating nanofibers with microfabricated structures. A microfabricated triangular tip, integrated with microfluidics, was used as the electrospinning source. The microfluidic coupling allows new possibilities for on-chip materials processing and functional nanostructure formation. We have used a similar microfabricated device as a source for mass spectrometry. The fabrication process for this device, with a triangular thin film tip, was described in detail in previous reports.[14,15] Briefly, a triangular-shaped polymeric film was lithographically fabricated and aligned at the exit of an embossed microfluidic channel. This tip acted as a wick and helped to establish a Taylor cone. The apex angle of the triangular thin film was 90° and the thickness was 3 μm.

The experimental configuration of the nanofiber deposition system is shown in Fig. 1(a). The electrospinning device was placed on a linear translation stage and the deposition distance between the tip and a counter electrode was 2 cm. A 300 nl/min polymer solution flow rate was created with a syringe pump, connected to the pipette reservoir by silica capillary tubing. A voltage of 3–4 kV was applied to the gold wire in the reservoir to establish a Taylor cone. Figure 1(b) shows a scanning electron microscope (SEM) image of trenches etched into a silicon wafer. The trench widths were 3 and 5 μm with a depth of 2 μm. The silicon counter electrode was attached to a speed-controlled rotating motor. The linear velocity of the counter electrode relative to the microfabricated electrospinning source was varied from 0 to 168 cm/s. We demonstrated the electrospinning process with polyethylene oxide (PEO). The polyethylene oxide polymer solution was prepared by dissolving PEO polymer (MW 100 000) at a weight concentration of 10% in a 50–50 mixture of de-ionized water and ethanol. The microfabricated electrospinning source was imaged by optical microscopy with an Olympus AX70 microscope, which allowed us to visualize the polymeric liquid on the triangular tip. A stable Taylor cone and liquid jet were observed at the apex of the triangular tip, as can be seen in Fig. 1(c).

We studied the deposited nanofiber diameter on a planar counter electrode without rotation. Figure 2(a) is a scanning

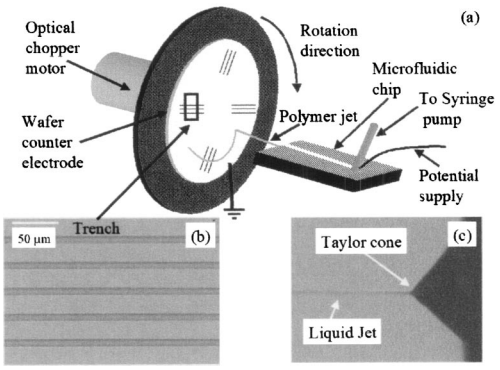

FIG. 1. System used for suspended nanofiber fabrication and for observation of liquid activity on the triangular tip source. (a) The electrospinning device was located 2 cm from the silicon wafer attached to an optical chopper motor. There was a fluid reservoir connected to a syringe pump. A gold wire was inserted into the reservoir and connected to the power supply. (b) Trenches etched on a silicon wafer, fabricated in a radial direction as shown. (c) A Taylor cone established on the triangular tip to form a polymer jet.

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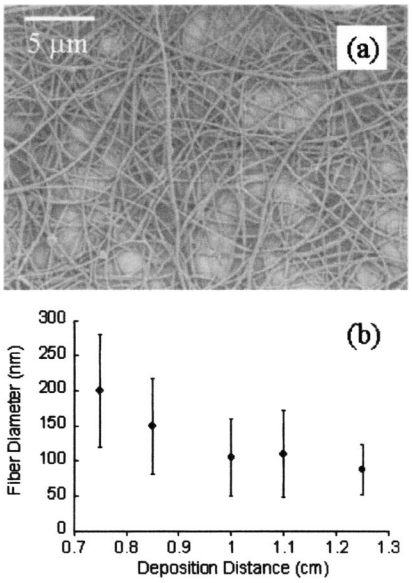

FIG. 2. (a) Scanning electron micrograph of nanofibers. They were fabricated by electrospinning PEO solution on the planar silicon counter electrode with the electric field of 2000 V/cm without rotational motion. (b) The nanofiber diameter as a function of deposition distance. The nanofiber diameter was averaged from 100 samples.

electron micrograph (SEM) of PEO nanofibers, fabricated on the silicon wafer with a deposition distance of 0.85 cm. With a stationary substrate, the fibers are randomly oriented. The nanofiber diameter as a function of deposition distance is shown in Fig. 2(b). The diameters of nanofibers were averaged from about 100 samples and standard deviations were calculated. When the deposition distance was less than 0.75 cm, membranes were fabricated rather than nanofibers. The averaged nanofiber diameter decreased from 202 to 105 nm as the deposition distance increased from 0.75 to 1.0 cm. Increasing the deposition distance to more than 1.0 cm did not greatly influence the average nanofiber diameter. All nanofiber depositions were conducted with a 2000 V/cm average electric field. It appears that once the fibers dried in transit between a source and a counter electrode, the nanofiber diameter was fixed and the fibers were deposited on the surface as a solid. If there was not enough time for the solvent to evaporate in transit (short deposition distance), nanofibers were spread on the surface to form membranes. From this result, we concluded that at least a 1.0 cm distance was required to deposit dry solid fibers. The smallest diameter of random nanofibers fabricated with this concentration was about 50 nm with 1.0 cm deposition distance. Our observed diameter dependence is consistent with what has been seen for conventional sources.[11]

We envision our type of source to be used to create organized structures of nanofibers. The effect of the rotational speed of the counter electrode on the morphology of nanofibers was studied. The linear velocities of the counter electrode were 0, 8.4, 42, 84, 126, and 168 cm/s. The deposition distance was fixed at 2.0 cm with an applied potential of 4000 V for 10–20 s. As the surface velocity increases the effect of the random motion becomes less significant and the orientation of the fibers is dominated by the linear driven motion. Figure 3(a) shows the SEM of nanofibers electrospun on the planar counter electrode with linear velocity of

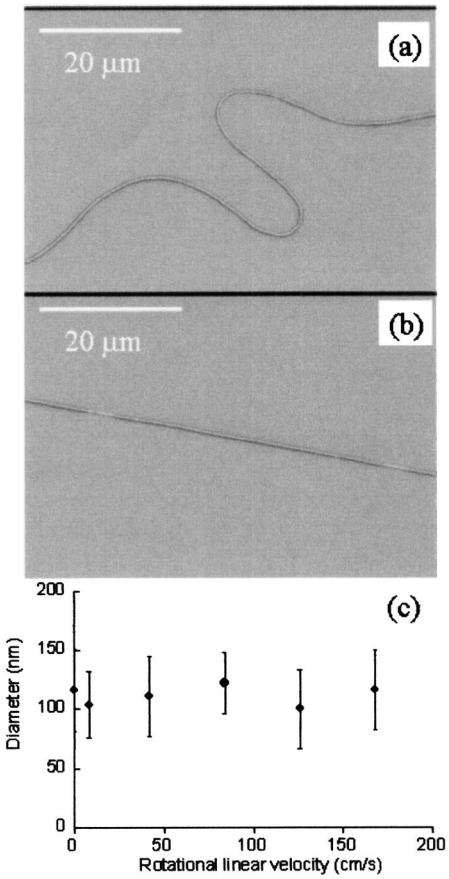

FIG. 3. Nanofiber fabrication by depositing on a rotating planar silicon wafer. The linear velocities of the counter electrode was (a) 42 and (b) 126 cm/s. (c) The average diameter of nanofibers as a function of rotational speed.

42 cm/s. Nanofibers were straightened by increasing the linear velocity. The SEM of straight nanofiber is shown in Fig. 3(b) (linear velocity of 126 cm/s). The diameter of nanofibers, averaged from 20 individual nanofibers, as a function of the rotational speed (from 0 to 168 cm/s) is shown in Fig. 3(c). The diameter of nanofiber does not significantly depend on the rotational speed. There is no orientation change of nanofibers observed with linear velocity of more than 126 cm/s.

As a demonstration of oriented nanofiber integration with microfabricated structures, we fabricated suspended straight nanofibers over etched trenches. The polymeric solution was spun on the silicon counter electrode with a linear velocity of 126 cm/s. Figure 4(a) shows the oriented nanofiber with about 150 nm diameter, fabricated over 3 μm wide trenches. Figure 4(b) is a SEM of about 140 nm diameter oriented nanofiber suspended over a 5 μm wide trench. The diameter of the nanofiber is constant over the trench.

In conclusion, we have fabricated oriented polyethylene oxide nanofibers using a microfabricated electrospinning device. The orientation of nanofibers was controlled by the linear velocity of the counter electrode relative to the source. We deposited suspended nanofibers oriented on etched silicon trenches. We feel that the use of electrospinning technology with a scanning source opens significant new possibilities for polymeric nanofiber creation and utilization. This ability to deposit nanowires oriented with respect to surface

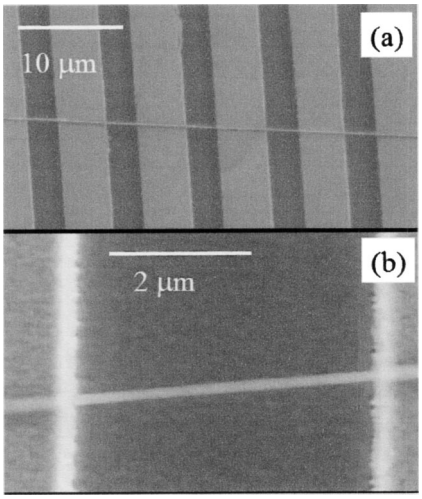

FIG. 4. Nanofiber fabrication by depositing on a rotating planar silicon wafer with etched trenches. The linear velocity of the counter electrode was 126 cm/s. (a) A suspended nanofiber, with a diameter of 150 nm, over a trench 3 μm wide and 2 μm deep. (b) A SEM of a 140 nm suspended nanofiber over a 5 μm wide and 2 μm depth trench.

electrodes or other nanowires could enable rapid formation of molecular electronic architecture.

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[1] X. Duan, Y. Huang, Y. Cui, J. Wang, and C. M. Lieber, Nature (London) **409**, 66 (2001).
[2] Y. Huang, X. Duan, Q. Wei, and C. M. Lieber, Science **291**, 630 (2001).
[3] Y. Cui and C. M. Lieber, Science **291**, 851 (2001).
[4] L. Liu, C. S. Jayanthi, M. Tang, S. Y. Wu, T. W. Tombler, C. Zhou, L. Alexseyev, J. Kong, and H. Dai, Phys. Rev. Lett. **84**, 4950 (2000).
[5] D. A. Walters, L. M. Ericson, M. J. Casavant, J. Liu, D. T. Colbert, K. A. Smith, and R. E. Smalley, Appl. Phys. Lett. **74**, 3803 (1999).
[6] Y. Zhang and H. Dai, Appl. Phys. Lett. **77**, 3015 (2000).
[7] J. Wang, M. S. Gudiksen, X. Duan, Y. Cui, and C. M. Lieber, Science **293**, 1455 (2001).
[8] J. Doshi and D. H. Reneker, J. Elects. **35**, 151 (1995).
[9] P. W. Gibson, H. L. Schreuder-Gibson, and D. Riven, AIChE J. **45**, 190 (1999).
[10] D. S. Katti, K. W. Robinson, M. A. Attawia, F. K. Ko, and C. T. Laurencin, Society for Biomaterials 28th Annual Meeting Transactions, (2002), p. 143.
[11] C. J. Buchko, L. C. Chen, Y. Shen, and D. C. Martin, Polymer **40**, 7397 (1999).
[12] I. D. Norris, M. M. Shaker, F. K. Ko, and A. G. MacDiarmid, Synth. Met. **114**, 109 (2000).
[13] A. Theron, E. Zussman, and A. L. Yarin, Nanotechnology **12**, 384 (2001).
[14] J. Kameoka, R. Orth, B. Ilic, D. Czaplewski, and H. G. Craighead, IEEE-EMBS Society Special Topic Conference on Microtechnologies in Medicine & Biology **12**, 62 (2002).
[15] J. Kameoka, R. Orth, B. Ilic, D. Czaplewski, T. Wachs, and H. G. Craighead, Anal. Chem. **74**, 5897 (2002).