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Microscale electrospinning of polymer nanofiber interconnections

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Polymer fiber interconnects were produced between microscale features on a substrate using only electrostatic forces. Electric-field-driven directed growth of nanoscale carboxymethylcellulose fibers was achieved between microscale droplets of a concentrated polymer solution. The fibers were studied using atomic force and scanning electron microscopy and were observed to emerge from the tip of conical protrusions formed at the surface of the droplets. The conical structures appear to be analogous to the characteristic Taylor cones formed in an electrospinning process and the process is interpreted as a microscale version of electrospinning requiring significantly lower driving potentials. © 2003 American Institute of Physics. [DOI: 10.1063/1.1594283]

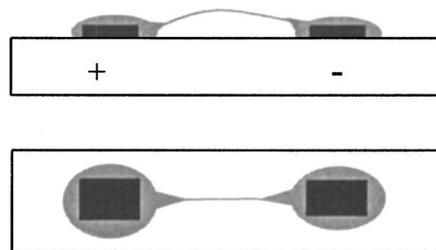
Polymer fibers form the basis of a wide variety of industries ranging from breathable, weather-resistant, and bullet-proof garments to telecommunications, structural engineering, and medicine. Polymer fibers are conventionally created by extruding a polymer melt through a spinneret and subsequently drawing the fibers as they coagulate. However, it is difficult to produce submicron diameter fibers using this conventional process and many emerging opportunities exist for high performance nanoscale materials and devices.

The recent focus on nanoscale engineering has revived interest in a radically different fiber formation technology known as electrospinning, wherein a polymer fiber is drawn from a solution using electrostatic instead of mechanical forces. The basic advantage of the electrospinning fiber formation process is that extremely small diameter, nanoscale fibers can be produced from a wide variety of polymer solutions.¹⁻³ The theoretical model for the electrospinning process has evolved over time and the fiber formation mechanisms have been described in several recent articles.⁴⁻⁶ Typically, an electrospinning apparatus consists of a hypodermic syringe or needle filled with a polymer solution and placed at a high (~ 15 kV) potential with respect to a ground plane. The sharp tip of the needle concentrates the electrostatic force and fibers emerge from the tip of a Taylor cone formed at the surface of the solution through a competition between electrostatic forces and surface tension. The fibers are collected at the counter electrode and typical electrospun structures consist of a nonwoven mat of fine fibers.

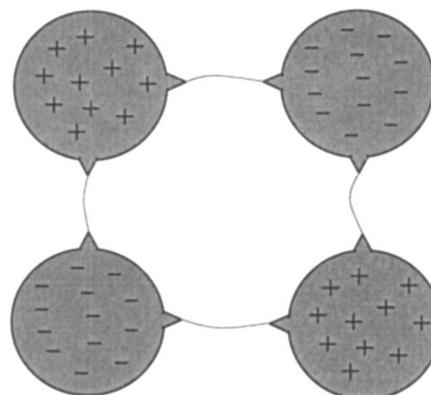
The physical laws of electrostatics that drive the conventional electrospinning fiber formation process are quite general and we have recently demonstrated that the entire process can be scaled to achieve directed nanoscale polymer fiber growth on the surface of a microchip without the need for high voltage, pumps, or needles. In fact, the fiber formation process actually appears to be favored at reduced dimensions due to electric field concentration effects. Thus, it should be possible to produce controlled nanoscale polymer

fiber structures and interconnections directly on the surface of a chip for numerous applications including; intrachip optical interconnections for the computer industry, chip-scale biocompatible fiber-based scaffolds, and highly sensitive micro-sensors.

Polymer fiber interconnections were produced in our laboratory using two methods. In the first method, illustrated in Fig. 1(a), a polymer was dissolved in a solvent and neutral, microscale droplets were airbrushed onto the surface of



(a)



(b)

FIG. 1. (a) Schematic diagram illustrating fiber formation between neutral droplets on oppositely charged electrodes. (b) Illustration of fiber formation between oppositely charged droplets applied to an insulating surface using electrostatic ionization.

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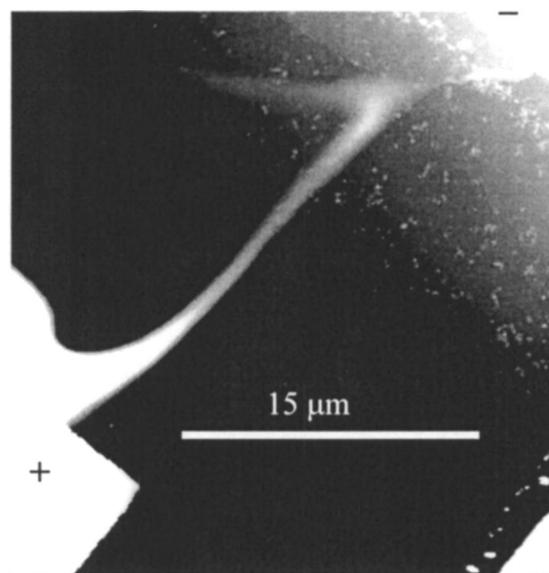
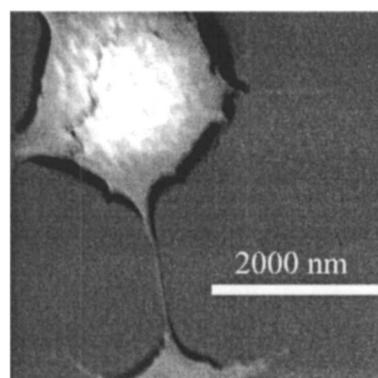


FIG. 2. AFM image of a single cellulose fiber formed between two interdigitated metal electrodes on a glass substrate.

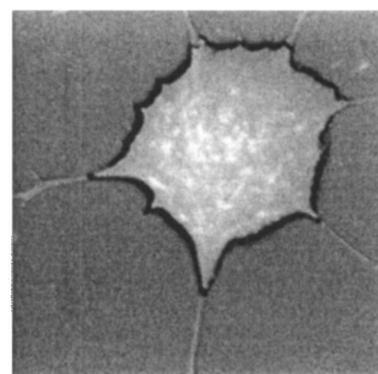
an interdigitated metal electrode on a glass substrate. A potential difference was applied between the electrodes and fibers were observed to form between droplets on alternating electrodes. In the second method, illustrated in Fig. 1(b), positively and negatively charged droplets were alternately sprayed onto an insulating substrate using electrospray ionization.⁷⁻⁹ The change in droplet polarity was achieved by switching the polarity of the high voltage power supply used to drive the electrospray process. In this case, nanoscale fibers were observed to form spontaneously between oppositely charged droplets without the need for the application of an external potential.

Figure 2 is an atomic force microscopy (AFM) image of a typical sub-micron diameter carboxymethylcellulose (CMC) fiber produced using method 1 between two metal electrodes separated by $15\ \mu\text{m}$ on the surface of a glass substrate. The CMC was first dissolved in a water/methanol solution at a concentration of 0.2 wt%. The solution was then airbrushed onto the electrode in the form of microscale droplets. The concentration of the polymer in the droplets on the surface is expected to be greater than the original solution concentration due to solvent evaporation and was not known in these experiments. A potential difference of 6 V was immediately applied between the interdigitated metal electrodes before complete evaporation of the liquid solvent. Due to the small spacing between the electrodes, this voltage difference produces an electric field magnitude on the order of 4 kV/cm, which is typical of the fields used in the conventional, macroscale electrospinning process. AFM images were obtained and compared to images from control samples produced in an identical process, but without an applied voltage. While numerous fibers were observed on the samples to which a voltage had been applied, no fibers could be found on the samples for which no external voltage was applied. Once established and upon removal of the potential difference, the dry, solvent-free polymer fibers were found to be mechanically stable and remained intact on the surface of the microelectrode.

We have also found that it is possible to produce similar



(a)



(b)

FIG. 3. SEM images of (a) a single cellulose fiber connecting two oppositely charged and (b) six cellulose fibers emanating from a single microscale droplet. The fiber diameters are all roughly 100 nm.

polymer fiber interconnections without the application of an external voltage. Figure 3(a) shows a single 100 nm diameter CMC fiber connecting two oppositely charged droplets, each approximately $2\ \mu\text{m}$ in diameter and Fig. 3(b) shows a single droplet with at least six individual fibers emerging from various locations around the circumference and connecting to oppositely charged droplets. CMC was dissolved in a water/methanol solution at a concentration of 0.01 wt%. Positively charged droplets were electrosprayed onto the polycarbonate substrate by applying a positive potential of 7.5 kV to the electrospray needle with respect to a ground plane established behind the substrate. Negatively charged droplets were applied to the polycarbonate substrate in the same manner by switching the power supply polarity. Scanning electron microscopy (SEM) and AFM images were obtained and compared to samples coated with both neutral droplets from airbrushing and single polarity droplets from electrospray. Fibers were only observed on the polycarbonate substrates coated with oppositely charged droplets.

Perhaps the most interesting result of this work is the natural extension of polymer electrospinning to microscopic dimensions for the production of nanoscale polymer fibers, interconnections, and scaffolds on the surface of a microchip. The fiber formation process is very simple and fast, does not require any special materials, chemistry, or equipment, and can potentially be applied to a wide variety of materials such as conducting, electroactive, photonic, and biocompatible

polymers. The images presented in Fig. 3 exhibit specific microscale features that are characteristic of an electric field driven fiber formation process. For example, each nanoscale fiber emerges from a small conical structure protruding from the surface of the droplet, which appears to be analogous to a Taylor cone. The cones are formed from a competition between the electrostatic forces and surface tension at a time before the solvent has completely evaporated. As the solvent continues to evaporate the viscosity of the droplet increases, preserving the electric field induced microstructure seen in Fig. 3.

The polymer fiber interconnects reported here were fabricated under a limited range of processing conditions (e.g., electric field magnitude, electrode spacing, and geometry) and more testing is necessary in order to determine the process window and overall generality of the approach. However, the conventional, macroscale electrospinning process has been successfully applied to a wide range of systems under various processing conditions and we, therefore, believe that the analogous microscale process described here will have similar generality. We are currently extending these initial studies to include other polymers, substrates, and ex-

perimental processing conditions. For example, other methods for the controlled application of the polymer solution onto a substrate are being investigated and applied to additional materials such as conducting and biocompatible polymers and even polymer composites and mixtures.

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