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Electrospinning of continuous aligned polymer fibers

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Electrospinning is a technique employed for preparing polymer fibers having diameters in the range of 10 μm–10 nm using high electrostatic field. In this letter, we report the formation of aligned polymer fibers, several centimeters in length, with separation between the fibers in the range of 5–100 μm. Achieving alignment is an important step toward the exploitation of these fibers in applications. We have employed about 4500 V and a separation distance of about 1–3 cm between the electrodes. Smaller distance between electrodes, we believe, provides better control on the formation of the fibers. © *2004 American Institute of Physics.* [DOI: 10.1063/1.1647685]

Fabrication of polymer fibers with diameters in the range of micrometers and nanometers has attracted the attention of several researchers in recent times all over the globe. This is in view of their potential applications in a wide variety of areas such as chemical sensors, electronic devices, filters, nanocomposites, smart textiles, etc.[1–3] Electrospinning[4–8] is one of the techniques, which has become popular, as it is simple and promising while at the same time posing tough challenges to scientists in different related fields.

Electrospinning involves the application of electrostatic force between polymer solutions or melt kept in a pipette or a syringe, and a counter metal electrode kept at a suitable distance. With the increase in electric potential the charged pendent drop of polymer solution formed at the tip of the capillary is deformed into a cone, known as "Taylor's cone." At a critical field when the force due to the electric field overcomes the surface tension forces holding the droplet, the solution starts flowing in the form of a charged jet. This jet moves toward the collecting counter electrode and while in transit most of the solvent molecules evaporate away and the different polymer strands in the jet separate out due to mutual repulsion, a phenomenon known as "splaying." When these reach the collecting electrode their diameters are in the range of a few micrometers to nanometers. Though the method of electrospinning has been known for a long time and is fairly straightforward, controlled production of aligned fibers reproducibly on a substrate remains a major challenge in this area. A few attempts have been reported recently toward aligning these nanofibers using electrostatic fields.[9,10] Aligning these fibers during electrospinning process is difficult because the method involves the application of very large electric voltages (10–30 kV) and large distances (15–30 cm) between the electrodes. Hence one may have to perhaps use even higher voltages with complicated electrode configurations to achieve focusing of the fibers for alignment. In addition, the fiber formation is critically dependent on several nonelectrical parameters such as viscosity, surface tension, concentration of the polymer solution and the choice of solvent, etc.

In this letter, a very simple approach toward aligning these fibers on flexible plastic substrates is described. Micrometer diameter fibers are obtained by applying voltages (<5 kV) much less than those usually employed. Consequently the distance between the tip of the capillary and counter electrode is also less (1–3 cm) to keep the electric field around 1000 V/cm as is usual.

Polystyrene (PS) (Mw = 2 50 000) obtained from LG Chemicals (India) and Polymethylmethacrylate (PMMA) (Mw = 120 000) from Aldrich chemicals, USA were used as such. Solutions with 15%, 20%, and 25% (w/w) of PS and PMMA were prepared with tetrahydrofuran and chloroform as the solvent, respectively. The solutions were freshly prepared by adding required weights of the polymer and stirred thoroughly for 6 h before electrospinning.

The experimental setup used for electrospinning is shown in Fig. 1. Polymer solution was held inside a 2 ml plastic disposable syringe (Dispovan, India). The inner diameter of the needle was 0.56 mm. A thin copper wire was inserted inside the syringe and connected to the positive terminal (A) of a power supply (5000 V). A thin stainless steel pin with sharp tip was used as the counter electrode (C). This pin was mounted vertically so that the tip of the pin lies exactly below the needle of the syringe (see Fig. 1). A 1-mm-thick polyester film cut in the size of 150 mm × 30 mm was used to collect the fiber by wrapping it around the cylindrical frame. This cylinder is attached to the axle of a 12 V dc motor (M2). This motor in turn was mounted with a slide-screw arrangement attached to a step-motor (M1) so that the whole assembly of the dc motor and the collecting substrate can be moved parallel to the axis of the cylinder. This helps in collecting fibers continuously on the plastic substrate while they are being formed. A control circuit for driving the step motor was built in the lab. The speed of the dc motor was controlled by a current source. The speed of the motor was measured for different currents using a photo-gate system prior to the experiment. During the experiment a pendent droplet was formed at the tip of the needle just before the application of voltage. The applied voltage was gradually

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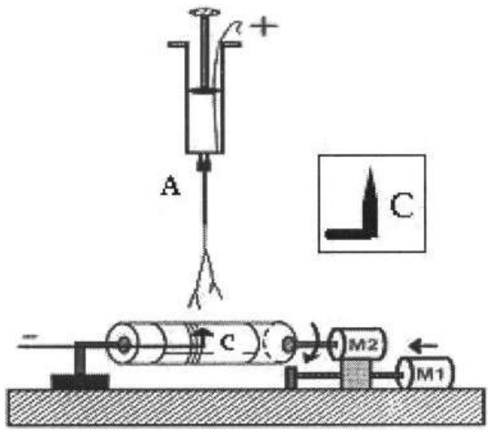

FIG. 1. Experimental setup for electrospinning. The polymer solution is taken in a syringe with a fine wire inserted as electrode (A). A steel pin aligned exactly with the needle of the syringe is used as counter electrode (C). A view of the pin (C) is shown in the inset. A 1-mm-thick flexible plastic film wrapped around an insulating cylinder attached to the axle of a dc motor (M2) is used as the substrate. The dc motor assembly is mounted on a plastic block. This block along with M2 and substrate can be moved linearly parallel to the axis of the cylinder using a step-motor lead-screw arrangement.

increased. With the increase in voltage the pendent droplet elongated to form the Taylor cone and at a particular critical voltage (~4.5 kV), a jet started from the tip of the cone.

Optical microscopy and scanning electron microscopy (SEM) were used for characterizing the fibers. Before taking electron micrograph pictures, the electrospun fibers were seen by optical microscope to confirm the formation of fibers. The SEM figures were obtained using Scanning Electron Microscope, Hitachi model S-3000H, after sputtering a gold film of thickness around 300 A on the substrate.

The SEM pictures (Figs. 2 and 3) show that very long and well-aligned fibers are formed having diameters in the range of 1–10 μm. Nanofibers are not formed mainly because of the low interelectrode distance and higher concentration of the solution. Though the applied voltage and the distance between the electrodes are reduced, the electric field was still around 1000 V/cm as those employed by others. The fibers are formed even with smaller applied voltage demonstrating that it is the field, which is responsible for fiber formation. This as such may not be a surprising result. However, if it could help in achieving better alignment due to greater control on the fiber formation then it may be useful. That is what is being demonstrated in this work.

In an effort to align the fibers, two techniques were employed simultaneously. One was to make use of a sharp needle as counter electrode instead of a large area metal plate. This may help in focusing the splaying fibers by the electric field lines converging on the needle. Similar efforts to focus the fibers were reported earlier with a sharp edged rotating disc.[9,10] Second, the substrate was made to rotate at high speed (~2000 rpm) using a dc motor. The speed was adjusted carefully so that the downward speed of the polymer jet and the linear speed of the substrate match. It was observed that when the speed of rotation of the substrate is around 1200–1500 rpm, continuous aligned fibers were obtained. This corresponds to a linear speed of about 2.5–3.0 m/s, which matches with the earlier observation.[6] The fibers obtained here were very long and straight and several centimeters in length. Fibers were also obtained in cross bar patterns by spinning the polymers in two mutually perpendicular directions on the same substrate.

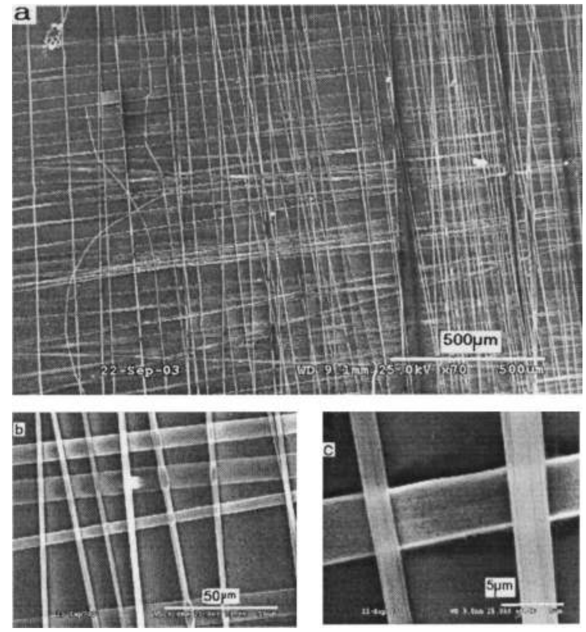

FIG. 2. SEM pictures of polystyrene fibers (25%w/w). The actual lengths of the fibers are in the range of several centimeters (~10 cm). The fibers are aligned and many of them are almost parallel to each other. The distance of separation among the fibers is in the range of 5–100 μm. Cross bar pattern of fibers have been obtained by forming long fibers in two mutually perpendicular orientations of the substrate. The diameters of the fibers are in the range of a few microns. Voltage applied=4.8 kV, distance between electrodes=1.2 cm. (b), (c) The fibers under higher magnifications (50 and 5 μm, respectively) are shown.

Figures 2 and 3 show SEM pictures of the PS (25% w/w) and PMMA (20% w/w) fibers, respectively. The PS fibers were obtained in cross bar patterns. The PMMA fibers are found to be discontinuous unlike the PS fibers. This is because the speed of rotation of the substrate was higher (~2000 rpm, corresponding to a linear speed of 4.2 m/s) compared to the speed of the jet. Figures 2(b) and 2(c), and 3(b) are also SEM pictures with higher magnification. Very good alignment can be seen in all the pictures. It can also be observed from Fig. 3(b), that the fibers have hollow tube-like structures, which collapse after formation on the substrate. This was also observed earlier by Koombhongse *et al.*[11]

The success in aligning the fibers in the present experi-

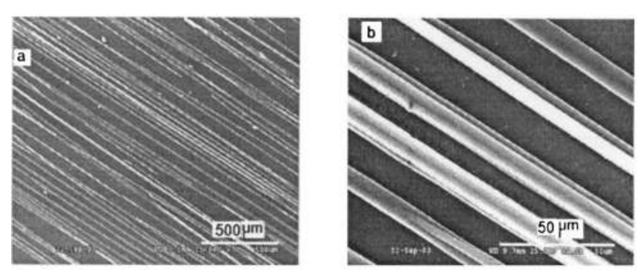

FIG. 3. SEM pictures of PMMA fibers (20%w/w). The fibers are cut at several places. This is because the speed of rotation of the substrate did not match with the speed of the jet in this case (see the text). Good alignment is achieved with very long fibers (10–12 cm). Voltage applied=4.8 kV, distance between electrodes=2.2 cm. (b) Under higher magnification (50 μm). Collapsed fibers in the form of tubes forming ribbon-like structures can be seen.

ment may be because of the following reasons: (1) the reduction in the interelectrode distance; (2) use of higher concentration of the solution so that very few solvent molecules are there, which can evaporate quickly during the small distance the fibers travel; and (3) use of a single sharp pin as counter electrode, which helps to focus the fibers due to focusing electric field configuration. The fiber diameters obtained are in accordance with the model proposed by Fridrikh *et al.*[12] It has been shown earlier that the diameter of the fiber depends on the surface tension, flow-rate, and electrical conductivity of the solution. Hence efforts are on presently to reduce the diameter of the aligned fibers to nanometer ranges by optimizing these parameters.

Long aligned (parallel and criss-cross pattern of) polymer fibers with diameters in the range of 1–10 μm were obtained on flexible plastic film substrate by applying a lower voltage (<5000 V). Fibers are several centimeters long and the spacing between the fibers is in the range of 5–100 μm. This we believe is a step forward in using these fibers in a variety of applications.

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