



ELECTROSPINNING: SHAPE AND ALIGNMENT CONTROL

Wenjie Liu*, Youqi Wang * and Ji Su **

*Dept. of Mechanical and Nuclear Engineering, Kansas State University,
Manhattan, KS 66506, USA

**NASA Langley Research Center, Hampton, VA 23681, USA

Keywords: Nanofiber, Electrospinning, Alignment

Abstract

Several approaches are presented in this paper. They allow us 1) to control the shape of deposited fabric precisely and 2) to achieve well aligned uniform fabrics with a controlled size. In order to control the shape of the fabric, two components are incorporated into the standard electrospinning process: a metal electric filed shield and a shaped hollow collector. The metal shield improves the electric field and thus reduces the chaotic nature of the spin-process. The shaped collector controls the shapes of the deposited fiber. Two approaches are used to control fiber alignment. In the first approach, a pair of cylindrical electrodes is used as collectors. A bundle of highly aligned fibers are produced between the cylindrical electrodes. By rotating these two cylindrical electrodes, a uniform deposited fabric with a consistent fiber alignment is produced. In the second approach, a programmable logic controlled electric field is applied during the spin process. During the process, fiber motion is controlled by the electric field direction. With the PLC controlled electric field, well aligned samples with large dimensions can be produced.

1 Introduction

In the 1930s, electrospinning was invented as a simple, versatile method to fabricate fibers [1]. Though it could produce fine polymeric fibers with diameters on the order of from less than 100 nanometers to a few micrometers, it drew little attention until recently. With the rapid development of nano-structured materials in the 1990s, interest in the electrospinning process revived due to its capacity to produce nano-fibers.

A conventional electro-spin setup includes a syringe, a needle and a collection plate. The syringe

contains a selected polymer. A high voltage DC power supply is applied between the needle and the collection plate. A force induced by an electrical field overcomes the surface tension of the polymer solution. A fiber is pulled out from the needle and deposited onto the plate. Generally speaking, however, the deposit process is chaotic. Fiber is randomly oriented on the collector.

Initial applications were mainly limited to the filtration and scaffold of tissues growth due to the disadvantage of the randomly oriented polymeric fibres. Although well aligned architecture nanofibers will have a wider application potential, it remains a challenge to control fiber orientation and fabric shape during the spin process.

In order to control the shape and size of deposit layers, one must accurately control the deposition position of spun fibers. To do this, Deitsel et al [2] used eight copper rings to generate an electric field, applying a centrifugal force to the spun fiber. As such, the deposit diameter was 4 times smaller than with standard electro-spinning. However, it is difficult to create a fabric with a uniform thickness and an accurate shape with their approach.

Efforts to align spun-fibers onto the collector divide into three categories. In the first category, high speed mechanical devices are utilized. Theron et al [3] and Katta et al [4] wound aligned nanofibers using high speed rotary devices. When the spun fiber is deposited onto a high speed collector, fibers are pulled in one direction. The weakness of this approach is the difficulty of matching mechanical device speed to fiber spin speed. Alignment quality is thus inconsistent. In the second category, parallel electrodes are used as deposit collectors. Li et al [5] first used two parallel electrodes to collect fibers. Both electrodes are connected to ground. When a spun fiber deposits on one electrode, a small electric field force pulls it to the other electrode. As such, a small aligned fabric is

produced. Similarly, Teo and Ramakrishna [6] used two steel blade knife edges to collect a small bundle of aligned fibers and then assembled the bundles to form a fabric. In the third category, Kim et al [7] and Sarkar et al [8] combine AC with DC power supplies in the electrospinning process. They connected multi-small paralleled electrodes or circular electrodes to the AC power supplies. Because AC power frequency is much higher than spun fiber oscillation, nanofiber alignment was achieved.

The research presented in this paper has two objectives:

- 1) To produce a deposited fabric with a predetermined uniform thickness and precise shape.
- 2) To achieve a well aligned uniform fabric with controlled size.

In order to produce a deposited fabric with an exact shape and thickness, a cone shield and various shaped collectors are used. In order to produce a fabric with well aligned fibers, two approaches are developed. In the first approach, two cylindrical electrodes are used to improve alignment. By slowly rotating the electrodes, a fabric is thus formed. In the second approach, a programmable logic controller is used to control the direction of the electric field. The electric field determines fiber motion. As such, a well aligned fabric is produced.

2 Shape Control

Control of deposit fabric shape during the electrospinning process is achieved through the introduction of two components: a metal cone shield and a shaped collector. The cone shield reduces fiber oscillation amplitude, the collector conforms a precise deposit fabric shape having a uniform thickness.

2.1 Standard electrospinning process

Fig. 1 (a) shows a standard electrospinning schematic drawing. The system includes a syringe, a stainless steel needle and a plate collector. The polymer solution is contained within the syringe. The needle is connected to a high-voltage DC power supply of 30 KV. The collector plate is grounded. An electric field is formed between the needle tip and the collector plate. The syringe pump continuously pushes the syringe at a certain speed to form a jet stream at the needle tip. The charged jet stream is stretched by the electric field force, causing it to become thinner and thinner. As the

solvent is evaporates, a fine fiber is produced. It deposits onto the collector plate.

In our experiments, the polymer solution was prepared by dissolving Polyethylene Oxide (PEO) at a weight concentration of 4% with a molecular weight of 106 into a solution. The solution is a mixture of 60% water and 40% ethanol. The other working parameters were: high voltage $V = 180$ kV, syringe rate $v = 0.2$ ml/h and distance between needle tip to collector $H = 12$ cm.

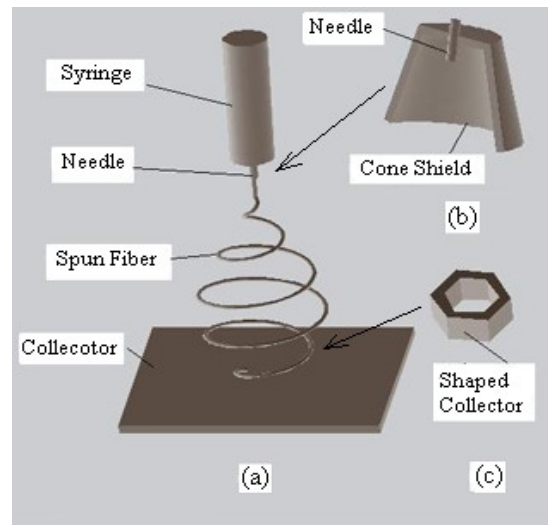


Fig. 1 Schematic drawings of (a) the standard method, (b) the cone shield method, and (c) the shaped collection method

2.2 Cone Shield

A cone shield is added to the system to control deposit position. The cone shield changes the electric field and reduces the deposit diameter. Fig. 1 (b) shows a schematic drawing of the cone shield, which is made of a metal material. It connects to the needle and has the same electric voltage as the needle. A piece of strongly absorbent Sontara cloth is plated on the top of the flat plate. Spun fiber deposit diameter can be adjusted by changing shield geometry, such as: cone shield angle, upper shield diameter and lower shield diameter.

Fig. 2 (a) shows an image of spun fiber deposit on the collector plate using a standard electrospinning process. Fig.2 (b) shows an image of spun fiber deposit with employment of a cone shield. Deposit diameter using the cone shield is less than 25% of deposit diameter using the standard electrospinning process. Random fiber oscillation amplitude is dramatically reduced.

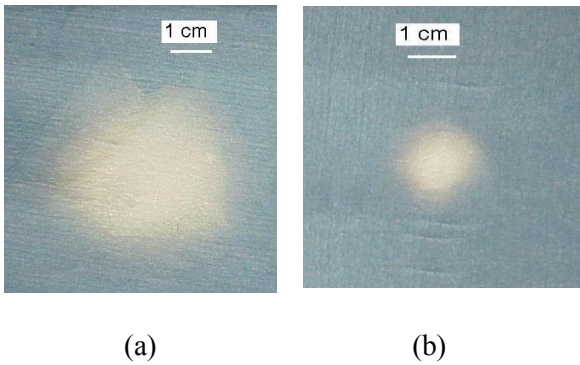


Fig. 2 Pictures of the mats of the spun fiber deposited from (a) the standard electrospinning and (b) the cone-shield method

2.3 Shaped Collectors

Fig 1 (c) shows a schematic shaped collector drawing. The shaped hollow collector is employed to replace the conventional collector plate. It is grounded. As the spun fiber deposits onto the collector, it brings a positive electric charge into the vicinity of the deposit area, which creates a voltage difference between the deposit location and other locations on the collector. As such, fiber is pulled to the other side of the boundary of the collector. During the spin process, fiber bounces between boundaries, forming a fabric with a predesigned shape and uniform thickness. The diameter of the collector used in this research ranges between 0.5-2". Three shaped collectors are used in our experiments: circular, hexagonal and square collectors.

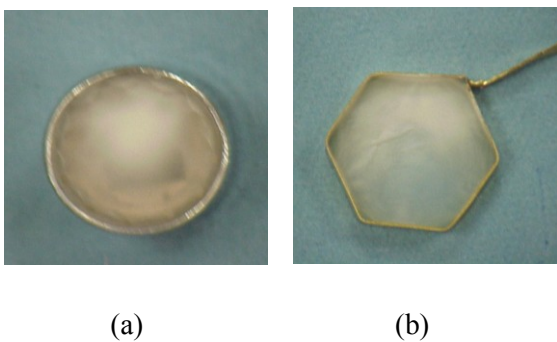


Fig. 3 Deposited nanofibers collected by the shape collectors with (a) circular and (b) hexagonal collectors

Fig. 3 (a) and (b) shows deposited spun fiber collected by using both the cone shield and the shaped hollow collectors. The ends of spun fiber are attracted to the collector upper surfaces and then are stretched by the forces between the positively charged fiber and the negative image charges on the

grounded collector surfaces. By controlling the total volume used in the spinning process, one can produce a fabric with predetermined shape and thickness.

3. Fiber Alignment

3.1 Coaxial Cylindrical Electrodes

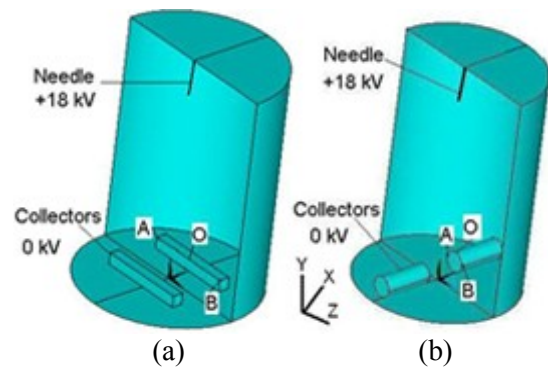


Fig. 4 The half of 3D FE model of (a) the PEM and (b) the CEM.

As aforementioned, the parallel electrode method (PEM) has been used in order to improve fiber alignment. Fig. 4(a) illustrates this approach. During the process, the fiber bounces back from one electrode to the other electrode, forming a fabric. Fiber orientation is perpendicular to the two electrodes. This approach has a weakness: Alignment quality is inconsistent along the two electrodes. For example, Fig. 5(a) is the fabric produced using the parallel electrode approach. The fiber is well aligned only in the small region close to the middle of the fabric. As arc shaped edges are generated, fiber is no longer perpendicular to the electrodes. In addition, the fabric is not uniform. It is thicker in the middle, with decreasing thickness as distance from the middle increases. Fig. 5 (a) was generated with 10 minutes of collecting time.

In this paper, the coaxial cylindrical electrode method (CEM) is employed to replace the parallel electrode method. Fig. 4(b) is an illustration of this approach. Two coaxial cylindrical electrodes replace parallel electrodes. Finite element analysis demonstrates that maximum electric potential occurs at the top point of the cylindrical electrodes. During the spinning process, the spun fiber travels between the top-point of one cylinder to the top-point of the other, forming an excellently aligned nanofiber bundle. Gaps between two cylinders range between

0.5-4 cm. Fig. 5(b) is the image of the fiber bundle produced by the coaxial cylindrical electrode method. Alignment is consistent and accurate. Fabric shown in Fig. 7 (b) was produced with 4 minutes of collecting time.

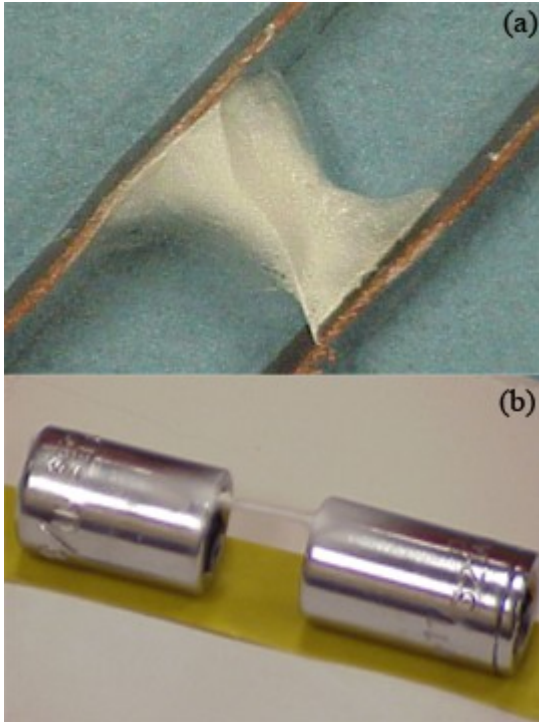


Fig. 5 Pictures of spun fiber collection from the (a) the PEM and (b) the CEM

3.2 Rotary Electrode Method

Using coaxial cylindrical electrodes, a well aligned fiber bundle is produced between the top positions of two cylindrical electrodes. By rotating the two cylindrical electrodes with identical angular velocity, a uniform and well aligned fabric can be produced.

Fig 6 shows a picture of the rotary electrode device. Two metal rings or electrodes fixed on a plastic sleeve are connected to the copper shaft using conductive wires through the sleeve holes. The shaft connected to the ground is supported by the slide bearings on the braces. A small DC voltage motor drives the shaft. The rotary speed of the electrodes is adjusted by the DC voltage. During electrospinning, the needle is placed in the middle of the two electrodes. The extending line of the needle passes through the coaxial line of the cylindrical electrodes. Under standard working conditions, aligned spun fibers are produced between the two electrodes.

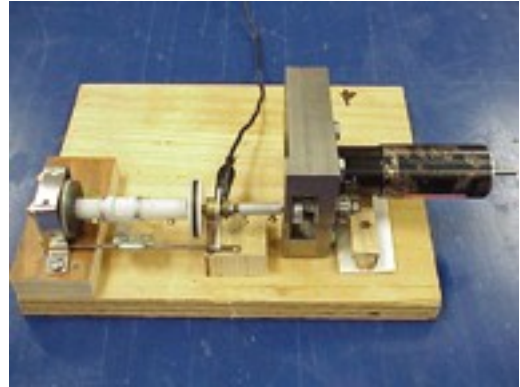


Fig. 6 Picture of the rotary electrode device with a pair of circular electrodes



Fig. 7 Photograph of the collection of spun fiber using the rotary cylindrical electrodes at 20 min of collecting time

Fig.7 shows a fabric generated using the rotary cylindrical electrodes. The diameter of the two electrodes is 1.5 cm. The fabric has a uniform thickness. A consistent alignment is seen from the ESM picture. Distance between the two electrodes ranges between 1-4 cm. Rotary speeds range between 0.5-15 revolutions per minute. The smaller is the rotary diameter, the better the achieved alignment. As the diameter becomes larger and larger, the coaxial cylindrical electrode method loses its advantage. Fiber alignment quality deteriorates. Further, if the rotating speed of the device is too fast, the two depositing points on the two respective electrodes will produce a small rotary angle and thus affect alignment quality.

3.3 PLC Controlled Secondary Electric Field

Though the coaxial cylindrical electrode method with a rotary device does generate a well

aligned fabric, two disadvantages are associated with it: 1) limited fabric size and 2) fiber orientation confined to one direction. In order to fabricate larger fabric and to design various fiber architectures, one must control fiber motion on a larger scale.

Electric field direction can control fiber motion. Therefore, a second programmable logic controlled electric field is applied to the spinning process. The apparatus is shown in Fig.8. Two power supplies are used. The syringe needle is connected to the first HV power supply. Two metal plates, placed on an insulator base, serve as electrodes. The two electrodes connect alternatively to a high voltage DC power source and a ground source through a programmable logic controller (PLC). As such, a parallel electric field is generated between the two electrodes. During the process, the PLC changes the direction of the electric field at selected frequencies. This forms an alternating dynamic electric field between two electrodes. As such, a well aligned fabric is produced. There are many paralleled, isolated, and small cylinders on a large isolated base between the electrodes. The spun fibers are aligned between the two electrodes and are supported by the small cylinders. The electrodes are rectangular blocks. Their top ends are 1~3 cm higher than the collecting plane formed by the small cylinder tops, so the inner planes of the electrodes provide an additional horizontal push or pull to the spun fiber. In this setup, the secondary electric field force is much stronger than the one in all previous methods. Therefore, fiber can travel over a large distance, and form a larger fabric. Fabric size can reach 20cm.

The electrospinning process is controlled using an Auto Direct PLC according to the operator's intent. Frequencies of the PLC and the HV relays can reach 50 Hz and 45 Hz, respectively. Relay maximum voltage is 30 kV. Refer to Fig.8. Each electrode is connected to two HV relays. Refer to Fig.8. One relay is switched to the high voltage (Pole 1 and 3) while another is switched to the ground (Pole 2 and 4). The connection sequence in one cycle is: (1) 1 and 4, (2) 2 and 4, (3) 3 and 2, (4) 2 and 4. In step (2) and (4), the high voltage electrode is switched to the ground in a very short time before the ground electrode is connected to the high voltage. This is because positive charges in the high voltage electrode need a certain time to discharge. If discharge time is insufficient, the voltage difference between the two electrodes is inadequate to direct the spun fiber. Discharge time is proportional to electrode volume. In experiments, minimum discharge time is about 0.01 and frequency f changes from 1 to 25 Hz. The spun fiber

reciprocates between the electrodes at the PLC frequency and deposits on the collector with a coaxially aligned pattern.

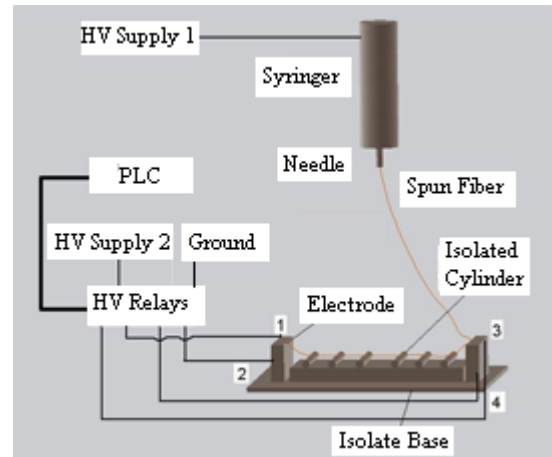


Fig. 8 A schematic drawing of PLC controlled secondary electric field approach

Two fabrics are produced using PLC controlled secondary electric field approach. 4% and 3% polymer solutions are used, respectively. The first fabric is produced using the following parameters:

- The first power supply: $V_1 = 18 \text{ kV}$
- PLC controlled power supply: $V_2 = 12 \text{ kV}$
- Spin speed: $v = 0.3 \text{ ml/h}$
- Distance between needle and collector base: $H = 12 \text{ cm}$
- Distance between two electrodes: $W = 18 \text{ cm}$
- Needle gage: $GZ = 22$
- PLC frequency: $f = 1 \text{ HZ}$.

The second fabric is produced using the following parameters:

- The first power supply: $V_1 = 10 \text{ kV}$
- PLC controlled power supply: $V_2 = 6 \text{ kV}$
- Spin speed: $v = 0.2 \text{ ml/h}$
- Distance between needle and collector base: $H = 6 \text{ cm}$
- Distance between two electrodes: $W = 10 \text{ cm}$
- Needle gage: $GZ = 23$
- PLC frequency: $f = 1 \text{ HZ}$

Fig. 9 shows pictures of the second fabric. It is the deposition of aligned nanofibers on the collector between the two parallel electrodes. It was produced in 15 minutes. The long spun fibers were formed between two electrodes and have a consistent alignment.



Fig.9 Deposition of aligned fibers in PLC controlled electric field

The diameters of the aligned spun fibers can vary in a wide range from tens of nanometers to a few micrometers in accordance with operating parameters. Fig. 10 shows ESM pictures for the two fabrics. The first fabric is shown in Fig.10 (a). The average diameter is about $2\ \mu\text{m}$. The second fabric is shown in Fig.10 (b). The diameters vary from 100 to 200 nm. One can see fiber diameter is sensitive to manufacturing parameters. Fiber diameter of the first fabric is 10 times that of the second fabric.

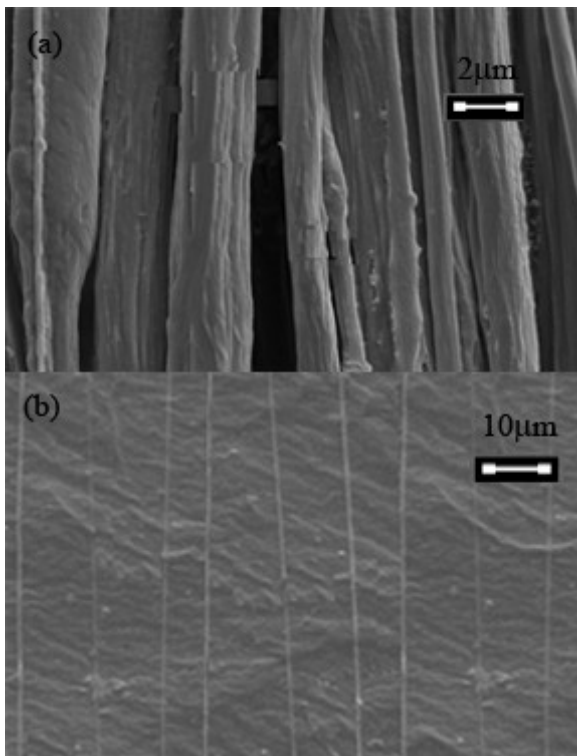


Fig. 10 ESM pictures of the aligned fibers from (a) the first fabric (b) the second fabric

4 Conclusions

In this paper, several approaches are developed to control fabric shape and fiber alignment in electrospinning processes. They include:

1. Two components are incorporated into the spinning device: a metal cone shield and a shaped collector. The metal shield reduces chaotic fiber oscillation and the shaped collector facilitates production of fabric with a uniform thickness and a pre-designed shape.
2. Two approaches are developed to achieve fiber alignment: a rotary coaxial cylindrical electrodes approach and a PLC controlled secondary electric field approach. With the rotary coaxial cylindrical electrodes, one can produce a small well aligned fabric with a uniform thickness. With the PLC controlled secondary electric field approach, one can control fiber motion on a large scale. As such, a large fabric with well aligned fibers can be produced.
3. With the PLC controlled secondary electric field approach, one can control fiber motion not only in a single direction. Rather, it becomes possible to fabricate a fabric with more complex fiber architecture. This will be explored in the future.

References

- [1] Formhals A., U.S. Patent No. 1975504, 1934
- [2] Deitzel J., Kleinmeyer J., Hirvonen J. and Beck Tan N. 2001 Controlled deposition of electrospun poly fibers *Polymer* Vol. 42 pp 261-72, 2001
- [3] Theron A., Zussman E. and Yarin A. Electrostatic field-assisted alignment of electrospun nanofibers *Nanotechnology* Vol. 12 pp 384-390, 2001
- [4] Katta P., Alessandro M., Ramsier R. and Chase G. 2004 Continuous Electrospinning of Aligned Polymer Nanofibers onto a Wire Drum Collector *Nano Lett.* Vol. 4 pp 2215-2218, 2004
- [5] Li D. and Xia Y. Electrospinning Nanofibers as uniaxially aligned arrays and layer-by-layer stacked films *Advanced Materials* Vol. 16 pp. 361-370, 2004
- [6] Teo W. and Ramakrishna S. Electrospun Fibre Bundle Made of Aligned Nanofibres over Two Fixed Points *Nanotechnology* Vol.16 pp 878-1884, 2005
- [7] Kim G. and Kim W. Formation of oriented nanofibers using electrospinning *Appl. Phys. Lett.* Vol. 88 pp 233101-233104, 2006
- [8] Sarkar S. and Tepper G. Aligned polymeric nanofiber arrays using biased AC electrospinning, *Fiber Society Conference, Knoxville, Oct., 2006*