Technote:

Electrospinning Carbon Fibers for Multiscale Fuel Cell Support Electrodes

Sean White, Department of Mechanical Engineering, Columbia University
sw2061[at]Columbia.edu
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Overview

Multi-scale fuel cell support electrodes promise improved surface area per volume for catalytic activity while maintaining conductive pathways. Electrospun carbon fibers provide a length scale in between the smallest (carbon nanotubes) and largest (carbon fiber paper) structures. This document discusses the process of electrospinning carbon fibers for this and related applications.

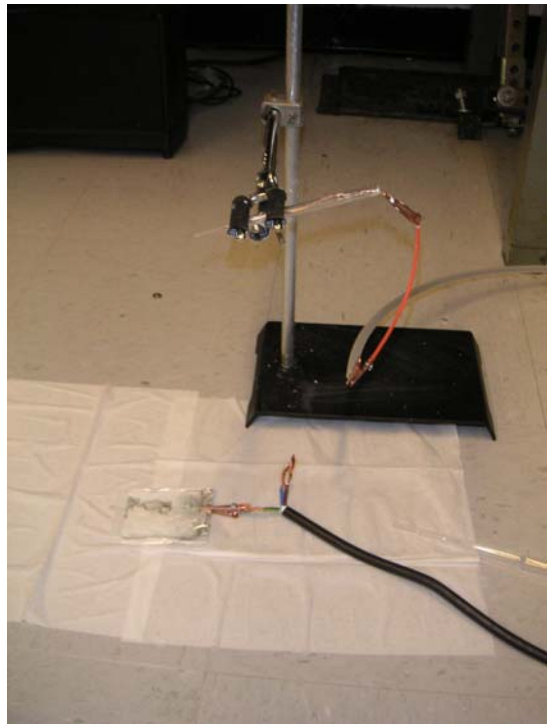

Figure 1

A solution of 100 mg/ml Polyacrylonitrile (PAN) in Dimethyl formamide (DMF) is prepared and placed in a small glass pipette held in a stand. The pipette is tilted so that the pipette tip is roughly 30 degrees off the vertical plane. This angle may change depending on the viscosity of the solution and should be set so that a round bead forms at the end of the pipette. The positive lead from a 30 kV Spellman high voltage source is placed in contact with the solution and the high voltage ground is connected to a 30 cm by 30 cm foil plate positioned 20-30 cm below the pipette tip. Once the electric field is present, a thin fiber is extruded from the tip of the pipette towards the ground plane and captured on a silicon chip, carbon paper, or other substrate. Continuous spinning of the fiber provides a fine mat of thin fibers. The fiber is then carbonized to complete the process.

Set-up

PAN and DMF were acquired from Sigma-Aldrich and mixed under a vented hood by slowly adding 1000 mg of PAN to 10 ml of DMF. In mixing the solution, care should be taken to dissolve as much of the PAN as possible. If large agglomerations remain, the particles may block the pipette or create discontinuities or spheroids in the spun fiber. A combination of sonication using a sonic horn and mechanical crushing large particles with a spatula followed by vigorous shaking was used to address this issue. Solution viscosity should appear much like hot molasses. Once the solution is prepared and the pipette is in position, it can be placed into the pipette for spinning.

The pipette is positioned in a stand, as in Figure 1, with the tip of the pipette 20 cm above the collection plate. Angle of inclination for the pipette plays a role in developing a good spun fiber. The pipette should be angled so that only a small round drop is formed at the tip of the pipette. The solution should not drip from the pipette but should be present at the tip.

The high voltage source should be appropriately grounded but the system should be as far as possible from any grounding source that is coupled to the high voltage ground. When the potential is very large, the spun fibers may try to extrude to physical locations that are also connected to ground and may miss the substrate. Care should be taken to secure the system so that the high voltage wires can not be dislodged and complete a circuit. As a safety precaution, always make sure the high voltage source is completely off before making contact with any part of the system.

Spinning

A Si chip or carbon paper is placed on top of the foil ground plane and used to collect the electrospun fibers. After the solution is placed in the pipette along with the positive lead, the voltage is slowly brought up to 20 kV until fibers are clearly seen collecting on the substrate. The electric field causes a polymer jet to form and accelerate towards the foil. As the solvent evaporates, the fibers should collect on the substrate. The spun fibers look like slowly forming cobwebs, as in figure 2.

Figure 2

The spun fibers may not immediately be visible. They can be identified by carefully waving a glass or other non-metal wand in front of the pipette to see if tiny strands appear. Care should also be taken to control the air around the experiment. A strong air current will keep strands from forming on the substrate and may blow stray strands into the lab. Do not inhale the electrospun fibers. Once the fibers have been spun they need to be carbonized in a two step process in a thermal furnace.

Carbonization

The pyrolisis process should be followed closely because the spun fibers will burn off if brought to high temperature in the presence of oxygen. First, the sample is heated to 250 C at a rate of 2 C/min and held for 3 hours in a Thermcraft tube furnace under air. This step removes any moisture from the sample and stabilizes the precursor fibers. Then, under Argon atmosphere, the sample is heated to 1000 C at a rate of 5 C /min and held for 1 hour before cooling to room temperature.

Carbon Nanotube Growth

As part of the multi-scale support electrode experiment, carbon nanotubes were grown on some samples of electrospun fiber deposited on a toray paper substrate. CNT growth was performed after pyrolization of the electrospun fibers using a CVD growth sytem that based on ohmic heating of the catalytic sites. Details of the CVD based ohmic growth system are discussed elsewhere. Growth was also done in tandem with the pyrolization process.

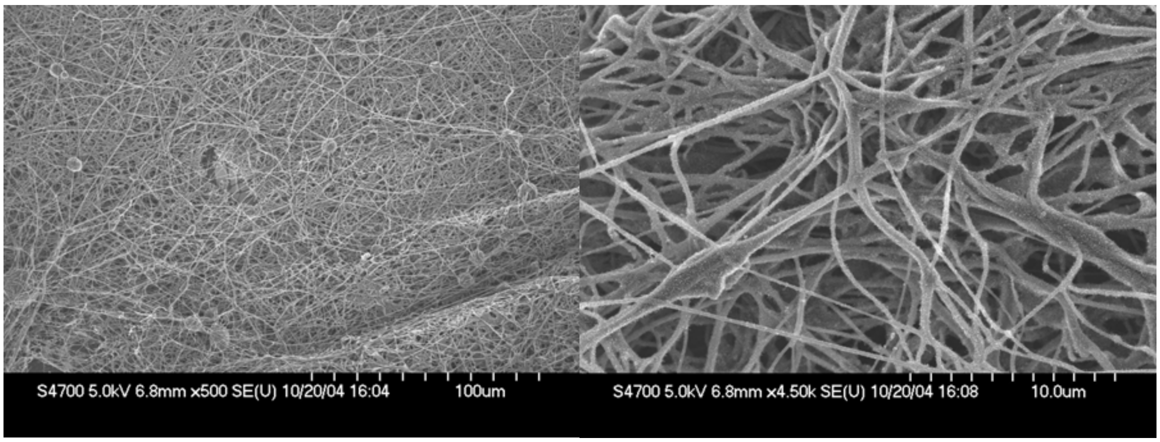

Figure 3

Characterization

Morphology of the electrospun fibers was examined under a Hitachi 4700 Scanning Electron Microscope.

Results and discussion

Prior to pyrolization, the fibers appear much like cobwebs on the substrate. Post pyrolization, the material looks brown and graphitic. Sample images are shown in Figure 3. Diameter varied but average diameter was around 500 nm. Figure 4 shows SEM images of the electrospun fiber on toray paper with carbon nanotube growth.

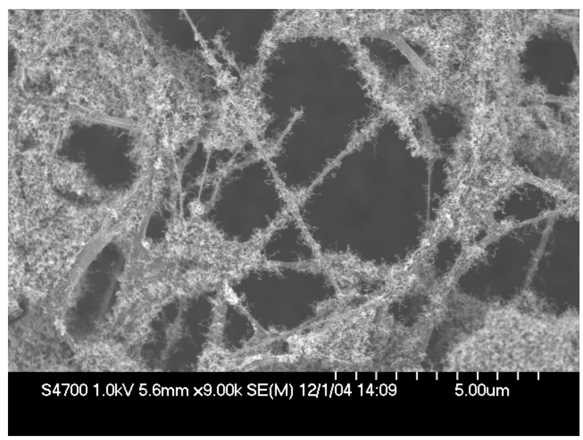

Figure 4

References

1. Doshi, J. and D. H. Reneker (1995). "Electrospinning process and applications of electrospun fibers." Journal of Electrostatics **35**(2-3): 151-160.

2. Li, D. and Xia, Y. “Electrospinning of Nanofibers: Reinventing the wheel.” Adv Materials 16 (1151-1170).

3. Hong Dong and Wayne Jones, tech report and conversation.