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Experiment and Simulation of Coiled Nanofiber Deposition Behavior from Near-Field Electrospinning

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Abstract- **Both experiment and simulation works were utilized to research the deposition behavior of single coiled nanofiber on silicon substrate from Near-Field Electrospinning (NFES). In the experiment process, when the collector moving speed (CMS) is compatible with electrospinning speed, straight line nanofiber can be collected. When CMS decreases, jet would step into whipping motion due to the imbalance charge repulsive force from landed nanofiber. As decreasing CMS, nanofiber in waved shape, single circle coil and multi-circle coil can be fabricated in turn. In order to improve the controlling technology, a computational model based on Maxwell viscoelastic theory was build up to analyzed the deposition behavior of single nanofiber. Simulation results show that CMS is the main controlling parameter influence the nanofiber deposition morphology: beads deposited in straight line, when CMS higher than 0.35m/s; beads deposited in waved shape, when CMS lies between 0.15m/s and 0.35m/s; beads in single-circle coiled can be gained, when CMS ranges from 0.08m/s to 0.15m/s; beads would deposit in multi-circle coil, when CMS lower than 0.08m/s. The effect of collector conductivity was also investigated by the computational modeling: the diameter of multi-circle nanofiber zone and distance between adjacent nanofiber coil increases with collector conductivity decrease. The calculated behaviors of nanofiber deposition are in good agreement with the experimental results, which is a good way to represent the motion behavior of charged jet and nanofiber.**

Keywords*- Near-Field Electrospinning, Nanofiber, Deposition Behavior, Computational Modeling*

I. Introduction

Electrospinning dates from 1934 [1], and attracts much attention all over the world with the development of nanotechnology [2]. Due to lots of special advantages [3], there are many potential applications of electrospinning technology and electrospinning nanofiber [4]. Near-Field ElectroSpinning (NFES) [5] is a new simple way to realize the deposition of single oriented electrospun nanofibers on planer substrate, which open a wider development space for the industrial application of electrospinning technology.

Electrospinning is a complex electrohydrodynamic process, the formation and motion of nanofiber is affected by many factors [6], such as electrical field, charge repulsive force, viscosity of polymer liquid. Electrospun nanofiber with some special characters of small diameter, light weight and high motion speed, which is difficulty to observed, measured and controlled during the electrospinning process. All of the precision position deposition, structure and morphology controlling of single electrospun nanofiber are difficult to be realized, which is still a big obstacle of the industrial application of electrospinning nanofiber [7]. At present, behaviors researching of electrospun jet motion and nanofiber deposition are the important aspects for the controllable deposition of electrospinning nanofiber [8]. The experimental and theoretical researching of the Taylor cone formation [9], bending instability of charged jet [10] and motion track of nanofiber [11] are the researching hotspots for electrospinning. The deposition behavior of single electrospun nanofiber attracts attention since the invention of NFES. Since nanofiber is difficult to be observed and measured during the electrospinning, theoretic and simulation models of single nanofiber deposition are required urgently for the control technology research of NFES. But few theoretic works have been done to describe the deposition process or morphology of electrospun nanofiber on substrate.

Coiled nanofiber is a representative structure for NFES nanofiber [12], which is a special way to study the motion behavior of nanofiber from NFES. In this work, both experiment and computational simulation were done to study the deposition behavior of coiled nanofiber from NFES. The process parameter effect and deposition controlling method of single electrospun nanofiber was also investigated.

II. Experiments

A Experiment Setup

The experiment system setup based on NFES is shown in Fig.1, including solid probe spinneret, high potential power supply and X-Y motion stage. Probe with tip diameter of 40μm used as spinneret in this experiment, discrete droplets of polymer solution were supplied in a manner analogous to that of a dip pen by immersing and pulling the tungsten electrode in to and out of the polymer solution. The anode of high potential power supply (DW-P403-1AC, Tianjing Dongwen High Voltage Power Supply Plant, China) was connected to probe, and the cathode was connected to the grounded silicon substrate. The applied voltage between spinneret and collector can be adjusted from 0 to 40kV. Collector can motion in X-Y direction, and the Collector Motion Speed (CMS) is in the range of 0~1m/s. 12%wt to 20%wt polyethylene oxide (PEO, average molecular weight = 300,000g/mol, Dadi Fine Chemical Co., Ltd., China) solutions in 60% vol /40% vol water/ethanol were electrospun.

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Electron micrographs of the nanofibers were obtained by using LEO 1530 Field Emission Scanning Electron Microscope and XL30 Field-Emission Environmental Scanning Electron Microscope (ESEM-FEG).

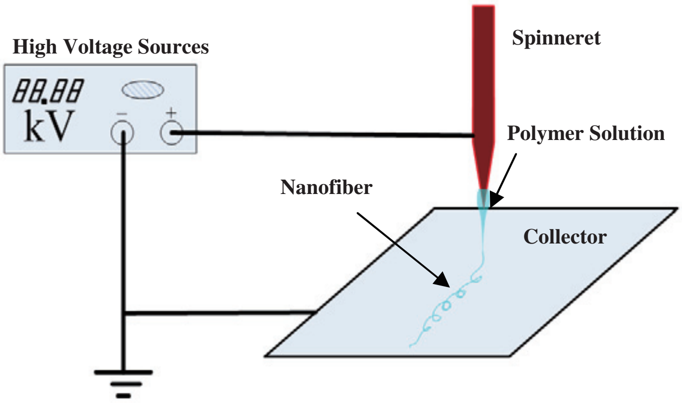

Fig 1. Schematic of experiment setup for Near-Field Electrospinning.

B Experiment Result

In the electrospinning process, the jet emanated from polymer drop is straight for a distance of several millimeters from the drop before bending [13]. In NFES, the distance between spinneret and collector is shorted to 0.5~3mm rather than 5~15cm in conventional electrospinning, by which the advantage of stable jet stage was taken to direct-write nanofiber. With the shorter distance, splitting of jet can also be overcome, and then single nanofiber can be electrospun. As the motion of collector, nanofiber can be deposited along the track of collector as shown in Fig. 2.

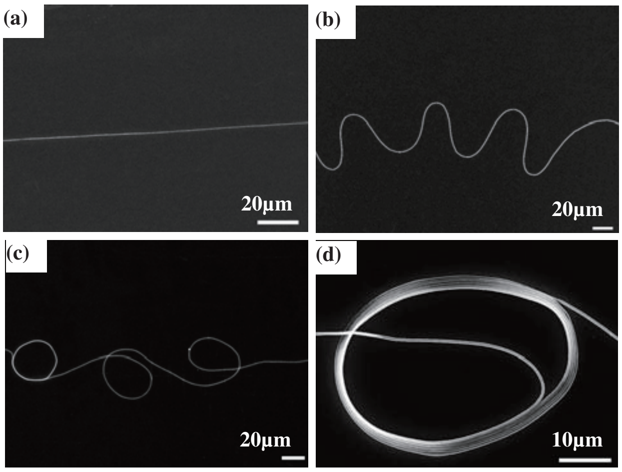

Fig.2. NFES nanofibers deposited on the silicon substrate. (a) Straight line nanofiber, CMS is 0.35m/s; (b) Waved shape nanofiber, CMS is 0.25m/s; (c) Single circle coiled nanofiber, CMS is 0.15m/s; (d) Multi circle coiled nanofiber, CMS is 0.05m/s. The polymer solution concentration, diameter of spinneret, spinneret-to-substrate-distance and applied voltage is 20%, 40μm, 1mm and 1.7kV respectively.

Though NFES can overcome the jet splitting, the motion and deposition of single nanofiber is still a complex process. The deposition behavior of nanofiber from NFES involves with many factors, such as CMS, electrical field, electrical charge, surface tension and position of spinneret. CMS is the major controlling process parameter that affects the morphology of single nanofiber. When CMS is higher than electrospinning speed, a strong drag force from spinneret applied on the jet to avoid the whipping motion of jet. Without whipping motion of charged jet, straight line nanofiber can be collected as shown in Fig.2 (a).

Drag force from spinneret decreases with CMS, and then charge repulsive force and electrical field force would play more important role in the deposition process of nanofiber. With the disturbance of electrical field force and charge repulsive force, charged jet would deviate from the straight line track and step into rotational motion, waved shape can be gained as shown in Fig. 2(b).

When CMS lower than electrospinning rate, falling jet is in relaxed state that would be disturbed easily by other factors. The charge repulsive force from the landed nanofiber plays the key role in the whipping motion of charged jet. There is a relax time scale for the charge delivered from the landed nanofiber to the ground, the charge density on the nanofiber decrease with the landed time. So the charge density of landed collector decreases with the arrow in Fig. 3. For the asymmetrical charge density of landed nanofiber, imbalance charge repulsive force would lead charged jet into rotation motion. With the whipping motion of charged jet, coiled nanofiber can be fabricated as shown in Fig. 2(c).

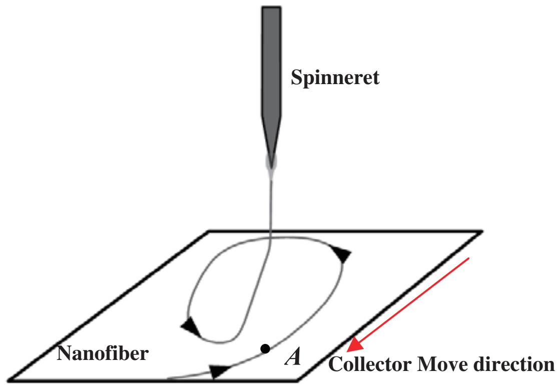

Fig. 3. Schematic of nanofiber deposited on the collector. Red arrow shows the collector move direction.

As CMS and drag force from spinneret decreased further, charged nanofiber has more relax time before deposited on the collector. Under lower CMS, more nanofiber would be deposited on the same zone. Due to the imbalance charge repulsive force and inertia of jet whipping, multi-layer nanofiber would be deposited in the concentric circle as shown in Fig. 2(d). For longer deposition time, most of charges on the nanofiber before point A in Fig. 3 had been transferred to the ground, and the charge repulsive force was smaller than that from the nanofiber later than point A. On the other hand, the nanofiber without charge is higher than the plane substrate, which changed the electrical field distribution. And then the electric field line point to the surface of nanofiber, which would guide the later nanofiber deposited along its top surface. Under the imbalance charge repulsive force and electrical field force [14], nanofiber would deposit layer by layer in the concentric circle and gain multi-layer coiled nanofiber.

III. Computational Simulation

A Computational Modeling

Due to high moving speed and small diameter of charged jet or nanofiber, it is difficulty to observe the deposition process of NFES nanofiber. A computation model is required to analyze the parameter influence and deposition behavior in the fabrication process of coiled nanofiber. Maxwell model [15] is one of class computational models to describe the behaviors of a material that have both viscous and elastic under stress, which has properties of both the solid and the liquid phase. Maxwell model has been utilized by many authors to describe the dynamic process from liquid jet to solid nanofiber of electrospinning [13, 16-18]. But few work focus on the deposition morphology of nanofiber on collector.

The diagram of the computational model is shown in Fig.3, the jet is supposed to be composed of a series of electrically charged beads and each one carrying the same mass $\boldsymbol{m}$ and charge $\boldsymbol{q}$. Every bead is considered to locate at the center of jet or nanofiber. Every two adjacent beads were connected by a viscoelastic Maxwell element, which contains a couple of series-wound spring and dashpot, the original length between two adjacent beads is $\boldsymbol{l_0}$. One new bead would create at the tip of spinneret, when the distance $\boldsymbol{d}$ between latest bead and spinneret tip is longer than $\boldsymbol{l_0}$. The charged bead was pulled downward by the electrical field force.

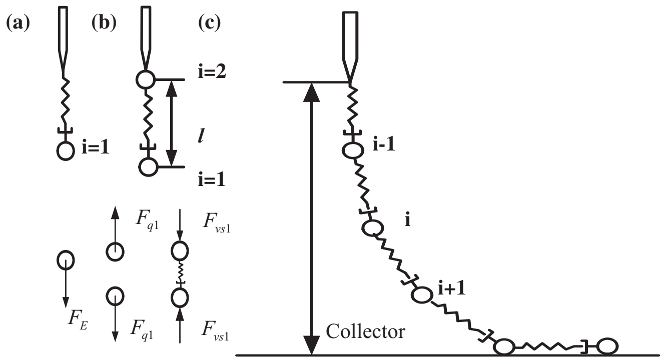

Fig.3. The diagram of computational model. (a) Charge bead moves downward driven by the electrical field force. (b) One new beads created at the tip of spinneret, when the distance $\boldsymbol{d}$ between the first bead and the spinneret. Charge repulsive force and viscoelastic stress force appeared from the adjacent beads. (c) When beads reach the collector, beads would stay at the deposition point. It is assumed that the bead strain between the spinneret and collector is continuous.

According to the Tip-Plane model [19], the electric field is given by Eq. (1).

$$E(X) = \frac{s \cdot C}{X(2s - X) + (s - X)r} \tag{1}$$

with

$$C = \frac{U}{\ln[2(s/r)^{1/2}]} \tag{2}$$

Where $\boldsymbol{U}$ is applied voltage; $\boldsymbol{S}$ is the distance between spinneret and collector; $\boldsymbol{r}$ is the radius of spinneret tip; $\boldsymbol{X}$ is the distance from the spinneret tip to the calculated position.

The distance between two adjacent beads can be calculated by Eq. (3).

$$l_u = \left((x_i - x_{i-1})^2 + (y_i - y_{i-1})^2 + (z_i - z_{i-1})^2\right)^{3/2} \tag{3a}$$

or

$$l_u = \left((x_{i+1} - x_i)^2 + (y_{i+1} - y_i)^2 + (z_{i+1} - z_i)^2\right)^{3/2} \tag{3b}$$

Where $(x_{i-1}, y_{i-1}, z_{i-1})$, (x_i, y_i, z_i) and $(x_{i+1}, y_{i+1}, z_{i+1})$ is the location of $\boldsymbol{i}$**-1**, $\boldsymbol{i}$ and $\boldsymbol{i}$**+1** bead. The original distance between two adjacent beads is supposed as 10^{-6}m.

The viscoelastic force acted on the charged bead is [16]:

$$\frac{d\sigma}{dt} = G\frac{dl}{l \cdot dt} - \frac{G}{\mu}\sigma \tag{4}$$

In Maxwell model, the viscoelastic stress is given as:

$$\dot{\sigma} = G\left(\varepsilon - \frac{\sigma}{\mu}\right) \tag{5}$$

Where $\boldsymbol{t}$ is simulation time; $\boldsymbol{G}$ is the elastic modulus; $\boldsymbol{\mu}$ is viscosity of polymer solution; $\boldsymbol{\sigma}$ the viscoelastic stress; $\boldsymbol{\varepsilon}$ is axial strain of charged jet.

The charge repulsive force between two charged beads in the falling jet is:

$$F_{qij} = -k\frac{q_i q_j}{l_u^2} = -k\frac{q^2}{l_u^2} \tag{6}$$

Where $\boldsymbol{q_i}$ and $\boldsymbol{q_j}$ is the charge of $\boldsymbol{i}$ and $\boldsymbol{j}$ beads, both of which are equal with the constant $\boldsymbol{q}$; $\boldsymbol{k}$ is coulomb constant.

As discussed above, when charged beads deposited on the surface of collector, the charge carried by the beads delivered to ground on a time scale of the order of $\tau = \xi/(4\pi\delta)$, where $\boldsymbol{\xi}$ and $\boldsymbol{\delta}$ is the effective electrical permittivity and conductivity of the collector. So charge in landed beads decrease with the deposited time $\boldsymbol{t_{land}}$, then the charge in landed beads can be given as a function of $\boldsymbol{q_{Lt}(t_{land})}$. Charge repulsive force between landed $\boldsymbol{y}$ bead and falling $\boldsymbol{x}$ bead is:

$$F_{qxy} = -k\frac{q_x q_y}{l_u^2} = -k\frac{q \cdot q_{Lt}}{l_u^2} \tag{7}$$

The difference charge in landed beads represents the imbalance charge repulsive force, which is the major factor leads to the formation of coil nanofibers. Combining the charge repulsive force from the falling beads and landed beads, the integrate charge repulsive force applied on every bead can be given as:

$$F_{qi} = -k\sum_{j=1}^{i-1}\frac{q_i q_j}{l_{ij}^2} - k\sum_{j=i+1}^{m}\frac{q_i q_j}{l_{ij}^2} = \sum_{j=1}^{i-1}F_{qij} + \sum_{j=i+1}^{m}F_{qij} \tag{8}$$

The momentum balance for beads can be given based on these force analyze:

$$m\frac{dv}{dt} = \pi a^2\sigma + qE + F_q \tag{9}$$

Where $\boldsymbol{v}$ is the velocity of bead; $\boldsymbol{a}$ is the radius of jet or nanofiber; $\boldsymbol{q}$ is the charge on every bead.

A numerical simulation program based on Four Bands Runge-Kutta method was made up to analyze the deposition process of charged nanofiber. For the high motion speed of jet during the electrospinning process, the 0.5~3mm distance between spinneret and collector takes less than 1ms for charged jet. Typical time step of 10^{-7}sec was chosen or the simulation model.

B Simulation results

According the experiment results, CMS and the time scale of charge transfer on the collector are the important parameters for the nanofiber deposition, which were discussed and analyzed based on the simulation program.

CMS is the main controlling parameter influence the deposition morphology of nanofiber, the effect of which was simulated firstly. Nanofiber deposition morphology under difference CMS can be gained based on the computational program, simulate results shown in Fig. 4. Red coils and blue lines refer the mass beads and Maxwell elements respectively in the simulation result. The distribution of beads and straight line represents the deposition morphology of nanofibers, and the distance between two adjacent beads refers the stretch of jet or nanofiber. As CMS decrease from 0.4m/s to 0.05m/s, nanofiber in the shape of straight line, wave, single circle coil and multi-circle coil can be gained. The relationship between nanofiber morphology and CMS is shown in Tab.1, of which simulation parameters are the same as that shown in Fig.4. Simulation results show that, beads deposited in the straight line when CMS higher 0.35m/s; the average distance between two adjacent beads is about 8.9×10^{-6}µm and that is stretched to 10 times than the original length as shown in Fig.4 (a). Beads would off the straight line tack and deposited in waved shape, when CMS ranges from 0.15m/s to 0.35m/s; the width of waved nanofiber is about 25µm, the average distance between two adjacent beads is about 9.4×10^{-6}µm in Fig.4 (b). Nanofiber was forced to deviate from the track of collector motion, which was stretched more than that deposited in the straight line. Due to the charge repulsive force, jet would step into rotation motion and beads deposited in coiled shape, when CMS lies between 0.08m/s and 0.15m/s; the width of coiled nanofiber is about 40µm, the average distance between two adjacent beads is about 2.4×10^{-6}µm in Fig.4 (c). Under the strong imbalance charge repulsive force beads would be lead to deposit in multi-circle coil, when CMS less than 0.05m/s; and the diameter of the multi circle nanofiber zone is about 30µm, the average distance between two adjacent beads is about 1.5×10^{-6}µm and deposition time scale of nanofiber is about 10ms during the simulation program in Fig.4 (d). For the lower drag force from the spinneret, tensile capacity of nanofiber decrease with CMS from waved shape nanofiber to multi-circle coiled nanofiber.

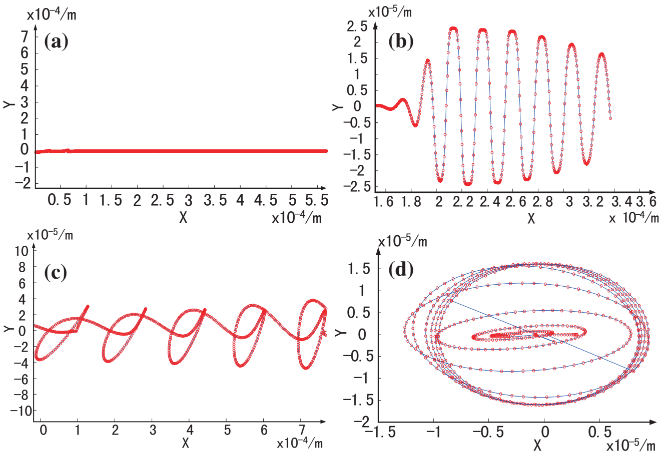

Fig.4. Simulation results from the computational model. (a) Beads deposited at a straight line, CMS is 0.4m/s. (b) Beads deposited in the wave-shape, CMS is 0.25m/s. (c) Coiled bead strain, CMS is 0.1 m/s. (d) Multi-circle bead strain, CMS is 0.05m/s. Concentration of polymer solution is $\boldsymbol{C}$=20%; viscosity of solution is $\boldsymbol{\mu}$=100 Pa • s; density of solution is $\boldsymbol{\rho}$=0.8537g/mL; charge density of bead is $\boldsymbol{q}$=10pC/m^3; the radius of bead is $\boldsymbol{a}$=10µm; the radius of spinneret tip is $\boldsymbol{r}$=20µm; the distance between spinneret to collector is $\boldsymbol{h}$=1mm; applied voltage is $\boldsymbol{V}$=1.7kV; effective electrical permittivity of substrate is $\boldsymbol{\xi}$=3.9; conductivity of the collector $\boldsymbol{\delta}$=0.435×10^{-5}S/cm.

Tab.1. Relationship of nanofiber morphology and CMS

CMS (m/s)	>0.35	0.15~0.35	0.08~0.15	<0.08
Nanofiber Morphology	Straight line	Waved shape	Single coil	Multi circle coil

Imbalance charge repulsive force from the leaded nanofiber is the main factor for the formation of coiled spiral nanofiber. Collectors with difference conductivity were utilized in the computational model program, and the effect of charge transfer time scale on the deposition morphology of nanofiber was discussed. Conductivity of collector decrease from Fig. 4(d), Fig. 5(a) to Fig. 5 (b), and the other electrospinning parameters keep constant in these three simulations. As noted above, charge transfer time scale increase with collector conductivity decrease, larger charge repulsive force would be applied on the falling nanofiber within the same deposition time. With smaller charge repulsive force, most of the nanofiber aggregate together nearby the same circle in the Fig.4 (d); but few coiled nanofiber aggregate together at one circle in the Fig.5 (a); furthermore every two adjacent nanofibers coils depart from each other in Fig.5 (b). Within the same simulation time, the distance between adjacent nanofibers coils and the diameter of multi-circle nanofiber zone increases with the conductive of collector decreases. The distance between two adjacent nanofibers is about 2-3.5μm in the Fig.5 (b), which is larger than the distance in Fig.4 (d) and Fig.5 (a). The diameter of multi-circle nanofiber zone is 28μm, 32μm, 80μm in Fig.4 (d), Fig.5 (a) and Fig.5 (b) respectively.

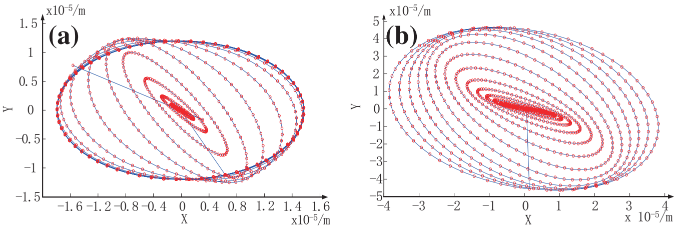

Fig.5. Simulation results of multi-circle bead-string structure on substrate with difference conductivity. (a) The effective electrical permittivity of collector is ξ=5.6, conductivity of the collector $\delta=1\times10^{-9}$S/cm; (b) The effective electrical permittivity of collector is ξ=11.9; conductivity of collector $\delta=1\times10^{-11}$S/cm. CMS is v=0.5m/s; Concentration of polymer solution is C=20%; viscosity of solution is μ=100 Pa • s; density of solution is ρ=0.8537g/mL; charge density of bead is q=10pC/m^3; the radius of bead is a=10μm; the radius of spinneret tip is r=20μm; the distance between spinneret to collector is h=1mm; applied voltage is V=1.7kV.

This computational model based on Maxwell theory has described well the motion behavior and deposition process of nanofiber from NFES. Computational simulation results were in good agreement with the experimental results, and reflect the similar effect of CMS and collector conductivity on the morphology of nanofiber.

IV. Conclusions

The motion behavior of nanofiber in coiled nanofiber from NFES has been studied by experiment and simulation research.

NFES nanofibers in difference shapes of straight line, waved shape, single coil and multi-circle coil on silicon collector can be gained by adjusting the CMS. The charge repulsive force from landed nanofiber is imbalance due to the charge transfer time scale, which is the main factor for whipping motion of charged jet in NFES. On the other hand landed nanofiber with less charge would be a guider for the later nanofiber. Under the imbalance charge repulsive force and the guide of landed nanofiber, nanofiber would be deposited layer by layer and multi-circle nanofiber coil can be gained.

The force analysis of NFES process was done and a computational model base on Maxwell theory was build up to simulate the motion behavior of charged jet or nanofiber. Nanofiber deposition morphology can be gained by the simulation program, which reflects the similar effect of CMS and substrate conductivity as experimental results. This simulation model is a good way to represent the motion and deposition behavior of nanofiber. The computational simulation is an important aspect for the control technology research of single nanofiber deposition.

Though the simulation of nanofiber deposition morphology has been experiment validated, computational model should be perfected further to include the effect of evaporation, rheology, and discharge.

Acknowledgment

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