ULTRA LIGHTWEIGHT MATERIALS FOR BIO INSPIRED MICROSYSTEMS

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Abstract

Ultra lightweight nanofiber fabrics play a vital role in the development of Microsystems. Polyacrylonitrile, polybenzimidazole and Nylon-66 polymer based ultra lightweight nanofiber fabrics were produced using electrospinning technique. SEM characterization showed that the diameter of Nylon-66 nanofibers varied from 50-300 nm. The average modulus and strength of Nylon-66 nanofiber fabric was 2.4 GPa and 154 MPa respectively. An attempt to build dragonfly's wing using carbon fiber as grid and electrospun fabric as skin was made. Three types of phenomenological dragonfly wings such as carbon fiber grid, electrospun Nylon-66 nanofiber fabric bonded-carbon fiber grid and commercial Nylon-6 film bonded-carbon fiber grid were made. The flexural stiffness to weight ratio of electrospun Nylon-66 fabric bonded-wing was 160% higher than that of commercial Nylon-6 film bonded-wing. This shows the potential application of ultra lightweight electrospun nanofiber fabric for building Microsystems.

1 Introduction

The nature has inspired many innovations; prime examples are aircraft and hovercraft. Birds and insects continue to inspire people to invent newer flying systems utilizing the advancement of materials and structural concepts. Insects have living and nonliving parts; they can fly, crawl, walk and jump. These concepts will be adopted for the micro autonomous systems. Multifunctionality is a common principle of biological structures. The cuticle of insect wings, for example, fulfills the different demands like high stability combined with low mass and aerodynamically favorable construction. Dragonflies have been widely investigated to study their aerodynamic properties [1-2] and build Microsystems. The dragonfly has amazing structural characteristics that provide exceptional flying and hovering capabilities. This fly can accelerate to 60 mph in 1 second from still position and can flap wings at 35 Hz while feeding in flight. The data in Fig. 1 illustrates that the four lifting wings weigh less than 5% of the dragonfly's gross weight. The sun-dried veins serve as beam members and the flesh as membranes that result in ultra lightweight wings with areal weight of 5g/m^2. A typical one-ply composite (125μm thick) weighs about 200g/m^2 (40 times heavier). Further examination of the fly (Fig.2) membrane reveals that it is made of smaller veins (<10μm dia) dispersed within the membrane [3]. Such architecture offers superior tear resistance than standard membranes. This attribute is essential in any material that we develop. Modulus of wing veins and membrane were reported to be 2.9 ±0.8 GPa and 1.0 ±0.5 GPa respectively [3-5]. These properties easily achievable with many polymers (3 GPa), but the challenge is fabrication of ultra thin membrane with reinforcing fibers/rods to achieve high tear resistance. The idea chosen was to produce ultra thin reinforced membrane that mimic the insect wings by electrospinning polymer system followed by heat fusing of fibers to achieve interlocks and superior mechanical properties. The spacing between the fibers is so close that it will act like a closed surface for all aerodynamic purpose. This process creates a very high specific surface area material that could be used as an advantage to bring in other functionalities. Therefore, the development of Microsystems needs entirely new materials technology than those used for conventional aerospace applications. Fig. 2 illustrates the features of North Carolina dragonfly and its wings.

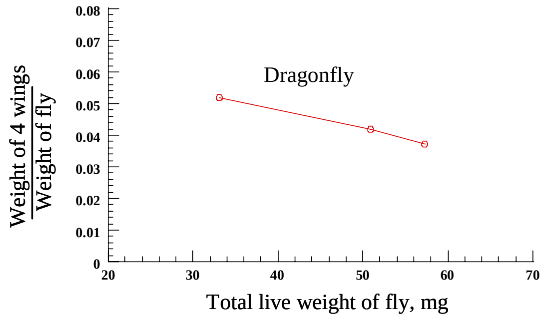


Fig. 1. Weight of wings versus total dragonfly weight

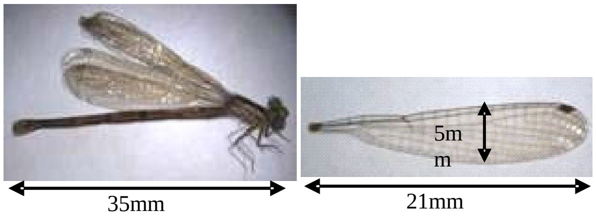


Fig. 2. Dragonfly and wings

The ARO's current MURI program provided us an opportunity to envision, conceptualize and start developing a new technology called electrospun nanofiberfabrics for building Micro Air Vehicles (MAV). Electrospinning is not new, and has been used in medical field. Only this team [6-7] and Zussman [8-9] are applying the technology to structural applications. Shivakumar and associates successfully developed a unique electrospinning setup and used the setup to produce electrospun nanofiber fabrics from various polymers. This paper describes the rotating drum electrospinning system, the materials developed, construction of phenomenological wings, measured microstructure, and mechanical properties of nanofiber fabrics and phenomenological wings.

2 Electrospinning Process

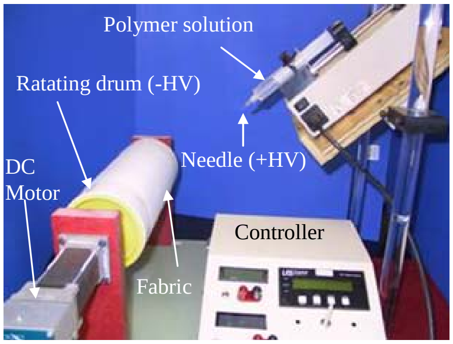


Fig. 3. Rotating drum target electrospinning system

Electrospinning was first documented in 1934 [10-12] and the process has been used for some medical applications as well as by other researchers interested in nanosized fibers. It utilizes an electrostatic force to spin fibers from a polymeric liquid. This liquid can be obtained thermally or by solvent solutions. In solution method, polymers are dissolved in a suitable solvent and stored in a syringe with a needle. The tip of the needle and collector are at a potential of tens of kilovolts. When the electrostatic force exceeds the surface tension of the polymer solution, the charges leave the droplet and drag the polymer to form fiber streams. The fiber streams become unstable and develop a whip-like motion that further elongates the polymer fiber resulting in a reduction in the fiber diameter before being collected on the charged collector screen. The polymer looses the solvent while traveling from the needle to the charged collector screen. Depending on the polymers and the operating conditions, the fiber diameter could range from few nanometers to a micron. Picture of electrospinning system with rotating drum target is shown in Fig. 3.

3 Material System and Process Parameters

3.1 Preparation of Different Polymers

Three potential polymers were selected for the study. Polyacrylonitrile (PAN) and Polybenzimidazole (PBI) were chosen because both are used as precursor in carbon fiber manufacturing and they can be oxidized to make it a thermoset resin. Nylon-66 was chosen as third polymer because of its high toughness and strength

Polybenzimidazole was obtained from Hoechst Celanese Corporation in Charlotte, North Carolina. The PBI polymer solution was prepared by dissolving dry PBI polymer in a mixture of LiCl (4% by weight) and N,N-dimethylacetamide (DMAc) under nitrogen gas at a bath temperature above the boiling point of DMAc for 4 hours with a refluxing condenser. LiCl was used to increase solution shelf life from days to several months. The PBI solutions were filtered to remove any residual polymer particles. The PBI solutions were prepared at 20 and 25% concentration (by weight) [7-14].

Polyacrylonitrile was purchased from Aldrich Corporation. The PAN polymer was dissolved in dimethylformamide (DMF) solvent. This mixture was vigorously stirred by an electromagnetically driven magnet at 60°C until it becomes a homogeneous polymer solution. Different concentrations of PAN solution (10, 15 and 20% by weight) were prepared using the same process [18-23].

Nylon-66 was purchased from DuPont Company (Zytel 101, MW=20,000 g/mol). Different concentrations (10-20% by weight) of polymer solution for electrospinning were prepared by using a solvent mixture of formic acid and chloroform with a ratio of 75/25 (v/v) [28]. The mixture was vigorously stirred by an electromagnetically driven magnet at ambient temperature until it becomes a homogeneous polymer solution [29-37]. Fig. 4 shows the steps involved in the production and analysis of electrospun polymer nanofiber fabrics. Electrospinning of Nylon-66 was carried out at different process conditions. Both randomly oriented and partially aligned nanofiber fabrics were prepared by rotating the drum at low speed (1000 RPM) and high speed (2500RPM) respectively [7].

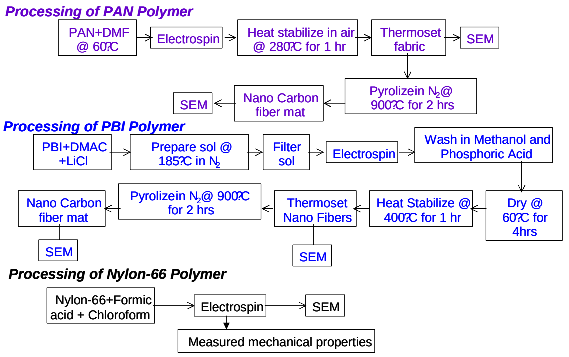
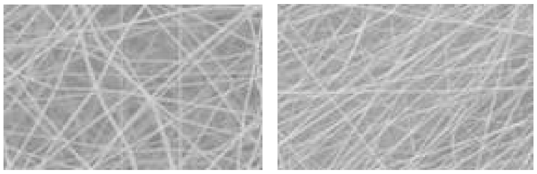


Fig. 4. Procedure for producing electrospun polymer nanofiber fabrics

3. 2 Morphology of Nylon-66 Nanofiber Fabrics



(a) Randomly oriented (150-300 nm)

(b) Partially Aligned (50-200 nm)

Fig. 5. Morphology of as-spun Nylon-66 nanofiber fabrics

The SEM images of randomly oriented and partially aligned Nylon-66 nanofiber fabrics are shown in Fig. 5. The diameter of Nylon-66 nanofibers varied from 50-300 nm. The fiber alignment increased by increasing the rotating speed of the drum.

3.3 Construction of Phenomenological Wings

An attempt to build dragonfly's wing using carbon fiber as grid and electrospun fabric as skin was made. The wing platform is about 102x28 mm. A dragonfly wing was enlarged 4 times and its features—frozen blood vessels by carbon fiber and membrane by Nylon-66 fabric/film were mapped. Fig. 6 shows the dragonfly wing (6.a), carbon fiber grid (6.b), and grids with electrospun Nylon-66 fabric (6.c) and commercial Nylon-6 film (6.d). The wing grid structure was constructed using 1K carbon fiber tow and EPON 9505 epoxy resin with 9554 Epicure curing agent. The resin was smeared to carbon fiber grid using a suitable brush. The grid structure was cured in air flow oven at 140°F for 2 hours duration. After curing, the excess resin was trimmed and Nylon-66 nanofiber fabric was bonded to carbon fiber grid structure as described above. For comparison, commercially available Nylon-6 film was also bonded to carbon fiber grid structure. The weights of the three manufactured wings were 215, 248, and 636 mg respectively. Electrospun Nylon-66 fabric weighed 1/13 of the commercially available Nylon-6 film weight.

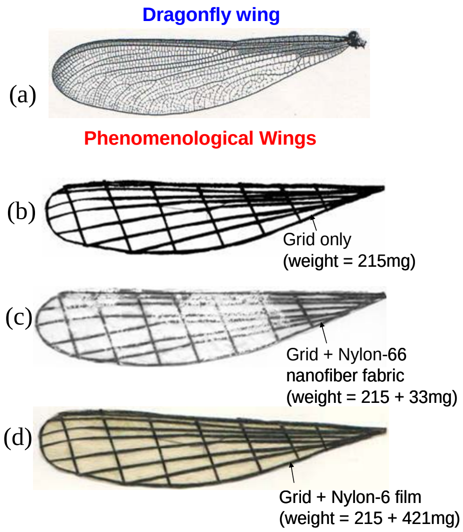


Fig. 6. Phenomenological wing configuration

4 Results and Discussion

4.1 Tensile Characterization of Nylon-66 Nanofiber Fabric

Main challenges in the submicron or the nanofiber fabric are the measurement of physical and material properties. The technology is not well developed. Furthermore, fibers are not well bonded, fabric is fuzzy and resulting in fuzzy definition for thickness. Therefore the areal weight which is assumed to be uniform is used to measure the thickness of nanofiber fabrics. The tensile behaviour of electrospun Nylon-66 fabric was tested using an Instron 4204 machine (Fig. 7) with a crosshead speed of 1.27 mm/min and at room temperature. For proper mounting and gripping on testing machine, Nylon-66 fabric samples were attached to a cardboard frame with 50 mm gage length and 6.2 mm width. Table 1 shows the initial modulus and ultimate strength of the nanofiber fabrics measured along longitudinal and transverse directions. The areal density of aligned and randomly oriented Nylon-66 nanofiber fabrics was 4.1 and 5.3 gm/m^2 respectively and the average thickness of the nanofiber fabric was 3.5 and 4.7 µm respectively.

Table 1. Tensile properties of Nylon-66 nanofiber fabrics

Speed RPM		**Average Modulus** GPa	**Average Strength** MPa
1000*	Longitudinal	1.1 (.04) [1]	62
	Transverse	0.8†	34
2500*	Longitudinal	2.4 (.27)[1]	154
	Transverse	0.3 (.02)[1]	17

[1] standard deviation
* spinning duration = 4 hrs.
† 2 samples

Fig. 7.Tensile testing setup for Nylon-66

The average modulus and strength of randomly oriented Nylon-66 nanofiber fabrics were 1.1 GPa and 62 MPa respectively in longitudinal direction and 0.8 GPa and 34 MPa respectively in transverse direction. For aligned fabrics they were 2.4 GPa and 154 MPa respectively in longitudinal direction and 0.3 GPa and 17 MPa respectively in transverse direction. The present values are higher than the values reported by Jung Dongwook [28] where the tensile modulus of electrospun Nylon-66 fiber varied from 23-108 MPa for different concentration of polymer. Also, for comparison, the bulk value of tensile modulus of Nylon-66 is 3.1 GPa and yield strength is 86 MPa [38].

4.2 Flexural Characterization of Constructed Wings

In this study, we examined the constructed phenomenological dragonfly wing's flexibility by measuring flexural stiffness (EI). Flexural stiffness is a composite measure of the overall bending stiffness of a wing using cantilever beam method. It is the product of the material stiffness (E, which describes the stiffness of the wing material itself) and the second moment of area (I, which describes the stiffness generated by the cross-sectional geometry of the wing). The flexural stiffness was measured in spanwise direction from wing base to wing tip.

Flexural stiffness of the phenomenological dragonfly wings was measured by applying a point force to bend the wing in spanwise direction. The applied force and the resulting wing displacement were used to calculate overall flexural stiffness (EI) using a beam equation. One end of the wing is fixed to supporting vice as shown in Fig. 8. A point force was applied using a pin at 70% of wing span. The pin slipped from the wing when it was placed too close to the edge. The resulting wing displacement was measured using a micrometer. The point loads were varied from 0 to 0.02 N and the corresponding displacements were measured. The distance from the fixed point of wing attachment to the point of force application was measured to determine the effective beam length (L).

Fig. 8. Flexural test setup for phemenological wings

Flexural stiffness is given by beam equation:

$$EI = {FL^3}/{3\delta}$$

Where F is the applied force and δ is the wing displacement at the point of force application (70% of span). This equation provides a measure of flexural stiffness over the entire beam length, and assumes that the beam is homogeneous, since the equation applies only to small displacements (less than 5% of the effective beam length) [39]. Fig.9 shows the load versus displacement curve for all the three different types of constructed wings. Table 2 shows the measured flexural stiffness of the three constructed wings. The Nylon-66 nanofiber fabric bonded carbon fiber grid was 1.16 times stiffer than the carbon fiber grid where as commercially available Nylon-6 film bonded grid was 1.14 times but weight of the nanofiber fabric is only 1/13 of the film. This demonstrates that for an equal flexural stiffness the electrospun nanofiber fabric is an order of magnitude (1/13) lighter than the film. The flexural stiffness to weight ratio of electrospun Nylon-66 nanofiber fabric bonded-wing was 160% higher than that of commercial Nylon-6 film bonded-wing (Fig. 10). This shows the potential application of ultra lightweight electrospun nanofiber fabrics for building Microsystems.

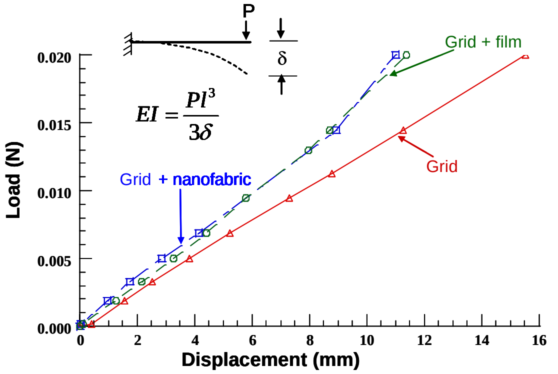


Fig. 9. The flexural response of phenomenological wings

Table 2. Flexural stiffness of phenomenological wings

Wing	Weight of wing (mg)	Flexural stiffness ($N-mm^2$)	Flexural stiffness to weight ratio ($N-mm^2/mg$)
Grid	215	140	
Grid + nanofabric	248	162	0.65
Grid + film	636	160	0.25

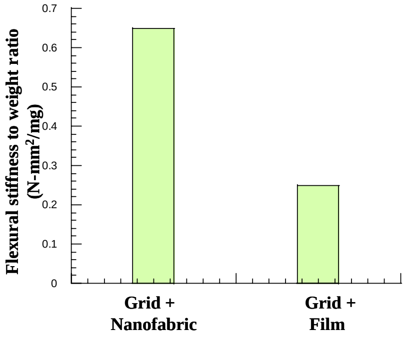


Fig. 10. Flexural stiffness to weight ratio of phenomenological wings

5. Conclusions

Polyacrylonitrile, polybenzimidazole and Nylon-66 polymer based ultra lightweight nanofiber fabrics were produced using electrospinning technique. Randomly oriented nanofiber fabrics were produced by rotating the drum at lower speed (1000 RPM) and aligned nanofiber fabrics were produced by rotating the drum at higher speed (2500 RPM). SEM characterization showed that the diameter of Nylon-66 nanofibers varied from 50-300 nm. Compared to randomly oriented Nylon-66 nanofiber fabrics, the tensile modulus and tensile strength of aligned Nylon-66 nanofiber fabrics were higher by 118% and 148% respectively in the longitudinal direction and lower by 62% and 50% respectively in the transverse direction. Three different types of phenomenological dragonfly wings were made: carbon fiber grid alone, electrospun Nylon-66 nanofiber fabric bonded-carbon fiber grid and commercial Nylon-6 film bonded-carbon fiber grid. The flexural stiffness to weight ratio of electrospun Nylon-66 fabric bonded-wing was 160% higher than that of commercial Nylon-6 film bonded-wing. This shows the potential application of ultra lightweight electrospun nanofiber fabrics for building Microsystems.

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