

Functional Fabric with Embedded Nanotube Actuators/Sensors

Project No.: M03-CL07s.

Competency: Materials

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1. GOAL STATEMENT:

Nanocrystals and nanostructures are often cited as candidate materials that can be engineered to exhibit enhanced or entirely new properties for use in different applications. The textile industry has also demonstrated a remarkable capability to incorporate both natural and manmade filaments into yarns and fabrics to satisfy a wide range of physical parameters that survive the manufacturing processes and are tailored to specific application environments. Along this line of reasoning and foreseeing the aforementioned textile industry's capabilities, this new seed-level NTC project proposed the development of next-generation functional fabrics (named here *electronic textile* or *e-textile*) utilizing single-walled carbon nanotube (CNT) composites. This exciting area of research in e-textile is motivated by discovery of bond extension in charged nanotubes (i.e., piezoelectric (PZT) effect in nanotubes). Our proposed nanotube-based fabrics, particularly after appropriate heat treatment, could become an enabling technology for a variety of macroscopic textile related applications such as actuation and shape modification of membrane structure, lightweight RF sensor system and power generation. In short, our objectives are: (1) nanotube-based fibers processing and fabrication, (2) development of CNT-based mono- and multi-filament yarns, and (3) design, development and control of functional fabrics with distributed actuation/sensing capabilities.

2. ABSTRACT:

The proposed nanotube-based fabric is prepared using a type of coagulation-based CNT spinning method in which CNTs are injected into a rotating bath of aqueous polyvinyl alcohol (PVA), washed and then slowly pulled from the bath. The spinning process will enable aggregation and alignment of the nanotubes in PVA. The resulting fibers are then processed into yarns for conversion into fabrics. This novel configuration shall translate the remarkable mechanical, electrical and thermal properties of nanotubes to a macroscopic scale in the proposed fabric. This annual report discusses our plans and approaches for the proposed e-textile, rather than emphasizing on the results since the project has just started (May 1, 2003). Some details and overview regarding our ongoing efforts for achieving the goals stated here are also provided in this report.

3. INTRODUCTION AND LITERATURE REVIEW

The development of e-textiles through the use of functional fibers could result in products with added value features that open entirely new market segments to the domestic textile industry. Current practice calls for fabrication of piezoelectric ceramic fibers by a rayon extrusion technique in which $\text{Pb}(\text{Zr}_x\text{Ti}_{1-x})\text{O}_3$ (PZT) ceramic materials are added to the fiber precursor and then spun into continuous ceramic fibers [1-3]. Although such PZT-based copolymers have found diverse uses in industrial applications such as ultrasonic transducers, hydrophones, microphones [4], and vibration damping [5], their low stiffness and electromechanical coupling coefficients (when compared to ceramics like PZT, for instance) have limited their use [6].

It is perceived that the intimate inclusion of CNTs in these fibers and ceramic matrix materials can completely change the properties of the base materials [7-14]. Current research on the production of CNT fibers and nanotube-polymer blends have achieved moderate levels of alignment of nanotubes [15, 16]. Methods of producing nanotube-polymer composite fibers and films have focused on melt-blending of PVDF-nanotube composites [17]. Other work on the formation of aligned nanotube fibers by re-condensing of surfactant stabilized nanotube solutions have also yielded good results [18]. The proposed area of research in e-textile stems from the fact that CNTs possess a very high aspect ratio with unusual electrical properties which make them suitable for distributed sensing and actuation applications. Termed “artificial muscles”, such actuators/sensors arrangement provides wonderful opportunities in functional fabrics due to their incredible strength and stiffness, with relatively low (~10 V) driving voltage [19-20]. These fibers have been shown to aid in direct conversion of energy from mechanical to electrical forms and vice-versa [21, 22]. This section provides an overview of the recent developments along this line.

3.1. Carbon Nanotube Synthesis, Characterization and Utilization:

The current research on carbon nanotubes (CNT) seems to be maturing from a stage of the study and characterization of the properties to the: i) application of electro-mechanical features in sensors and actuators design, ii) the integration of nanotubes in polymers to produce conducting fibers for smart fabrics, and iii) the application of the mechanical properties of the nanotubes to passively improve the properties of materials.

Gommans et al. [23] have described a method of producing aligned nanotube fibers using an electrophoretic self-assembly process. Raman spectroscopy of the fibers has confirmed the alignment of the nanotubes along the growth direction of the fiber. The method consists of drawing out a commercial carbon fiber electrode out of a suspension of single-walled CNT (SWCNT) in N, N, dimethylformamide (see Fig. 1). The SWCNTs are attracted to the positive carbon electrode and a dendritic structure of nanotubes is found to form around the carbon electrode in the dispersion. As the electrode is removed at a slow rate from the suspension, a bundle of nanotubes, in the form of a fiber of a couple of micrometers thick, is found to form on the end. The length of the nanotube fiber is only limited by the travel of the vice that holds the initial carbon fiber, and the availability of nanotubes in the suspension. The effects of the viscosity and surface tension of the surfactant and dispersion media on the ability to produce the aligned yarns and the contribution of these factors to the limiting velocity of drawing the yarn has not been studied. It may be possible to improve the yield of this process by analyzing how the choice of different dispersion media and surfactant affect the process

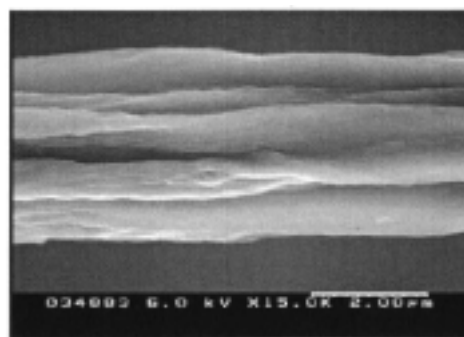
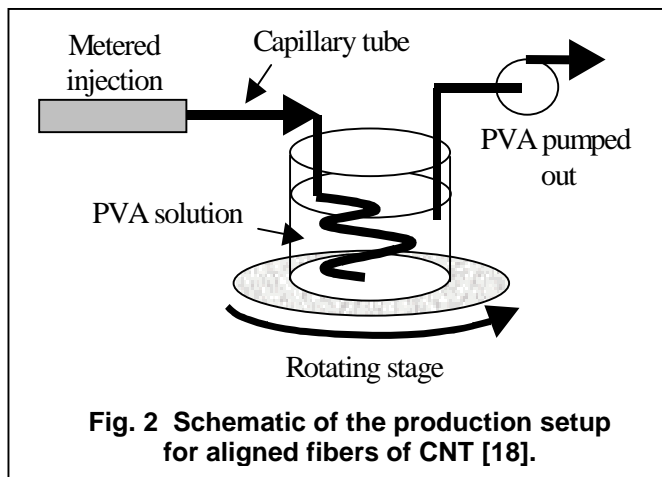


Fig. 1 SEM image of CNT fiber produced by electrophoretic drawing method [23].

A promising method to produce fibers of aligned nanotubes involves the orientation of nanotubes as a two-phased dispersion of nanotubes, in which a polymer is forced out of a capillary and then re-

condensed in a co-flowing polymer stream [18]. In this method, nanotubes are sonicated in an aqueous solution of sodium dodecyl-sulphate (SDS). The SDS surfactant mitigates the tube-tube interactions which can enable the formation of a nearly homogenous dispersion of the nanotubes in the solution. The dispersion is injected into a co-flowing stream of poly-vinyl alcohol (see Fig. 2). The orientation of the nanotubes in the fibers was confirmed by TEM and SEM imaging of the fibers, and manifested itself in the anisotropic characteristics of the fiber.



By using a modified technique based on the principle used by Vigolo et al. [18], Baughman and his team were able to produce long aligned fibers of nanotubes [24]. The process consists of introducing SWCNT dispersion into a co-flowing stream of PVA in a cylindrical pipe thereby causing the agglomeration of the SWCNTs into a ribbon. The flow rates are adjusted to give a fiber that had PVA within the matrix of the gel in order to give suitable strength to the fiber to be wound on a spool. This fiber is then unwound and passed through a series of washing stages to remove the excess PVA. Though the washing process never completely removes the PVA from the fiber, the small quantities of PVA in the nanotube fiber is found to enhance the load transfer between the nanotubes – possibly by the surface interaction of the nanotubes with the PVA. The process also has high yield rates of up to $70 \text{ cm}\cdot\text{min}^{-1}$, which is well suited to mass production of nanotube-polymer composite fibers.

3.2. Nanotube-based Fibers and Yarns Proposition:

Recently, continuous yarns of up to 30cm lengths of pure carbon nanotubes, with a diameter of about $200\mu\text{m}$, have been obtained by drawing them out of super-aligned arrays of carbon nanotubes [16]. The process is similar to the production of silk from a silk cocoon. It is also reported that the tensile strength of the yarn and electrical conductivity (after the yarn was used as a filament in a vacuum chamber) were increased. The discovery of the increase in the electrical conductivity of the yarns after the annealing treatment is important as it produces yarns of conductivity of up to 13% higher than conventionally produced yarns. Since there already exist methods to produce vertically aligned arrays of carbon nanotube yarns, this method can quite easily be adopted to produce carbon nanotube yarns which can be utilized in smart textile materials.

In a new process, Zhu et al. [15] were able to directly synthesize long ropes of nanotubes using an optimized CVD process in a vertical furnace. The process entailed the catalytic pyrolysis of n-hexane containing ferrocene and thiophene. The process was reported to produce long ropes of SWCNT at rates of up to $0.5 \text{ g}\cdot\text{hour}^{-1}$ and a purity of up to 95%. This is much better yield than claimed by other methods of SWCNT nanotube fabrication. The tests conducted also show that the tensile strengths of the nanotube fibers produced by this method were almost 5 times higher than that of nanotubes produced by other methods. This would yield nanotube-based ropes that could be utilized as passive materials in fabrics with very little processing effort.

The last two methods are the only ones that can produce pure nanotube fibers or yarns without much processing of the carbon nanotubes. However in terms of yield of yarns of nanotubes, and the possibility of adapting the process to manufacture yarns on a large scale, the methods suggested by Baughman et al., [24] and Zhu et al., [15] are the most promising methods that can be possibly improved to give nanotube yarns with potential of readily being woven into fabrics.

3.3. Nanotube-based Functional Fabrics Proposition:

Some studies have also focused on the production of functional fabrics and textiles by combining the properties of polymer materials with the nanotubes properties. Studies carried out so far have involved the melt-blending and electro-spinning of carbon nanotubes with polymer materials such as Poly-methyl-methacrylate, Poly-flouro-cyclo-butane and Poly-vinylidene-difluoride. Ko et al. [25] have demonstrated a method of production of nanotube-polymer composite yarns by electro-spinning solutions of polyacrylonitrile (PAN) and dimethylformamide (DMF)-nanotube dispersions. The SWCNT-PAN fibril was then carbonized and graphitized to get carbon fibers reinforced by SWCNTs. TEM images of the SWCNT-PAN fibril show the agglomeration of the nanotubes in the polymer matrix.

Seoul et al. [26] have described the electro-spinning of PVDF with nanotubes dispersed in DMF. The authors have shown experimentally the increase of conductivity of the fibers to be higher than that of pure PVDF by a factor of 4. This fact, along with the piezoelectric capabilities of PVDF, gives the possibility of utilizing the fiber in the form of embedded actuators and/or sensors in smart fabrics. The sensors could be used to sense pressure, force or deflection. Moreover, large-scale macroscopic actuation could possibly be achieved through the cumulative effects of the small deflections caused as a response of the material to externally applied voltages. Along this line, Xing [27] has studied the effects of nanotube loading on the piezoelectric properties of the PVDF-copolymer-nanotube composite. It is found that at low poling voltages, the response of the composite is not as good as pure PVDF. This has been attributed to the interaction of the nanotubes with the polymer matrix creating difficulties in the change of dipole direction of the crystalline polymer matrix. The higher required poling voltages suggest that the nanotube-polymer matrix interactions were affecting the orientation of the PVDF.

The development of reliable and repeatable methods of production of nanotube-polymer composite fibers is likely to lead to the utilization of such devices into intelligent textiles. One of the current requirements in the field of active fibers is the production of conducting fabrics that are also flexible and strong [28, 29]. Nanotube fibers have been proven to be highly torsion resistant and possess a higher strength than tough spider silks and steel. The high aspect ratios of the nanotubes imply that they are able to impart good electrical conductivities in polymer blends at low nanotube loadings and such materials have a low percolation threshold. Conductive fibers could also be used in cloth patches as antennae. As demonstrated by Winterhalter et al. [30], antennas woven into textile materials require less power for operation as compared to regular portable antennas. Such antennas could be used in civilian needs like RF identification tags woven into textile material as well as in battlefield communications antennas.

Dhawan et al. [31] have described the utilization of conductive fabric in the production of woven fabric-based electrical circuits. Though their work deals with the utilization of metallic conducting wires in the production of flexible wearable electrical circuits, the properties of nanotube-based yarns could be leveraged to produce more robust systems which are far lighter and more flexible than metallic wires (see Fig. 3). Post et al. [32], have described applications and methods of integration of conductive yarns into clothing and have dealt with the integration of conventional IC's and the utilization of fabric-based components as part of wearable computing devices. As a step in the direction of creation of nanotube-based electronic components for fabric applications, super-capacitors woven into a textile fabric has been reported [24]. The capacitors were shown to have capacitances and cycles life comparable to commercially available super-capacitors. Since nanotubes are also known to have effects similar to piezoelectric materials (when exposed to strains), such materials could be integrated into structures to produce sensors that monitor the stresses in structures.

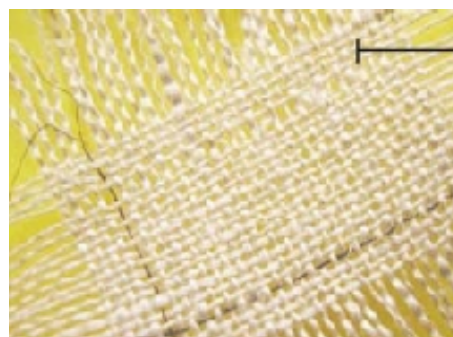


Fig. 3 Nanofiber super capacitors embedded into textile fabric [24].

3.4. Macroscopic Functional Materials Comprised of Carbon Nanotubes:

The early macroscopic form of single-walled CNT, called bucky paper, was obtained by drying purified nanotube suspensions. Such papers and sheets made of nanotubes have been utilized to create electromechanical actuators. However, the properties of such actuators are clearly not optimized given the random orientation of the functional particles in the plane of the sheet. Furthermore, the strength of these fibers is many orders of magnitude less than that of a single nanotube. This is mainly due to the weak Van der Waals and mechanical inter-locking forces between the nanotubes and the mechanical interlocking of the nanotubes. Although such forms of nanotubes will probably not find much practical applications, the conceptual design and system level integration may give insights into the capability of macroscopic actuation using nanotubes.

4. ONGOING EFFORTS AND RECENT DEVELOPMENTS

By incorporating highly distributed, discretized (yarn-based) active elements in woven (or knitted) fabric, both local and global properties can be altered to arrive at a “smart” textile configuration. In order to arrive at the proposed e-textile configuration, the following steps (as identified in the “Goal Statement”) have been targeted.

4.1. Nanotube-based Fiber and Polymer Processing and Fabrication:

Under this task, piezoelectric polymer fibers from single-walled carbon nanotube composites are being currently fabricated. Through a recent major initiative by Clemson University as well as PI, we have, so far, been able to synthesize CNTs in a very controlled and predictable method (depicted in Fig. 4 is our CNT synthesis and fabrication station housed in our recently-established Smart Structures and NEMS Laboratory at Clemson University, <http://www.ces.clemson.edu/ssnems/ntc.htm>).



Fig. 4 Easytube™ CNT synthesis and fabrication station housed in Smart Structures and NEMS Lab (acquired by both PIs and Clemson Univ. resources for feasibility study during the course of M03-CL07s).

4.2. Intelligent Control-based Nanotube Processing and Fabrication Proposition:

It is recognized that the astounding mechanical, electrical, chemical and thermal properties of nanotubes utilized in the proposed smart fabrics are highly dependent on their geometry and structures. Therefore, in order to fully realize the potential of such fascinating nanostructures in such e-textile applications (e.g., distributed sensors and actuators comprised of functional nanotubes), it is crucial to fabricate nanotubes with desired geometry and structure in a “controlled” growth environment. Specifically, we have started to utilize nonlinear and intelligent control techniques to regulate the selected nanotube processing state variables (e.g., reaction temperature and gas flow rate in a CVD) via combination of software and hardware modifications. That is, the influence of such nanotube processing state variables on the most vital and crucial aspects of nanotubes (e.g., length, diameter, yield, growth rate and structure) can be systematically controlled to spawn desirable properties with regard to nanotube fabrication, and thereby, maximize the nanotubes’ ability to function at their maximum potential for the proposed e-textile application.

In order to enhance the performance and precision accuracy of the produced nanotubes, a quasi on-line control experiment scheme is proposed (see Fig. 5). An interesting feature of such proposition is the ability to tune the nanotube growth conditions (such as gas flow rates and temperature profile) in order to make better nanotubes with desired characteristics. This iterative procedure is facilitated through a design decision making stage and based on an intelligent nonlinear control synthesis. In practice, one may interpret the macroscopic characterization and control experimental results and, depending upon its suitability, alter the nanotube growth conditions by designing a trajectory for selected process variables.

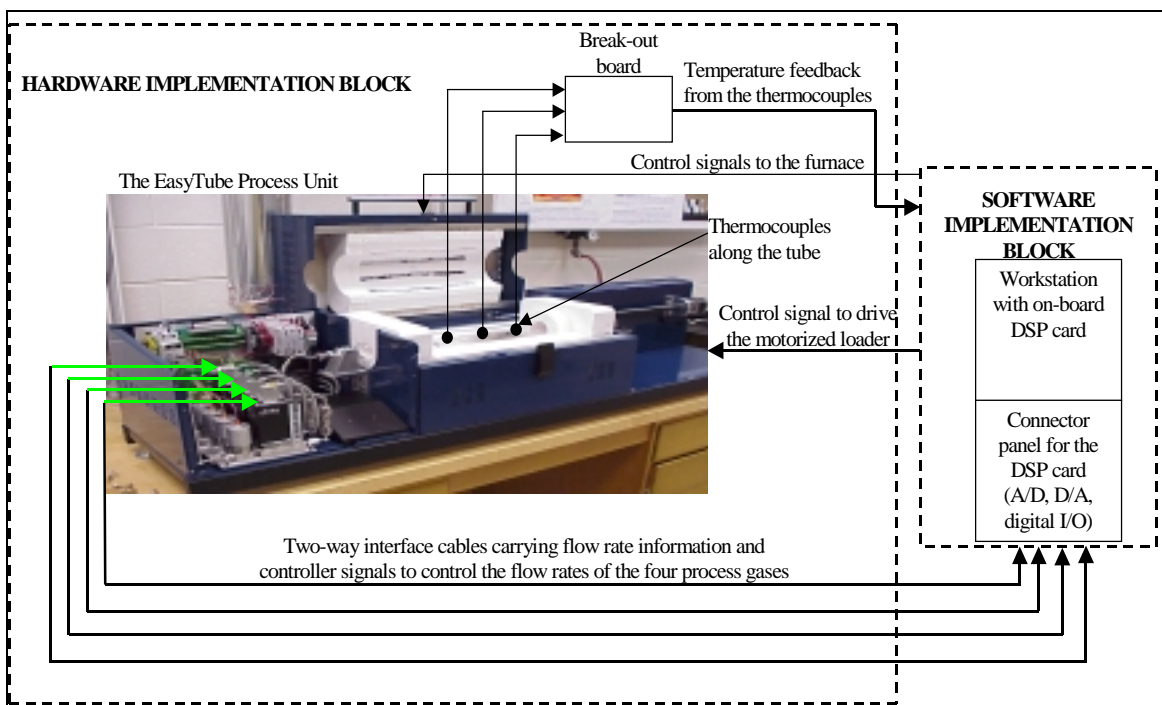


Fig. 5 Schematic diagram of the proposed intelligent control-based enhanced nanotube synthesizer via a CVD (inner photo) housed in the Mechanical Engineering Smart Structures & Nanoelectromechanical Systems Laboratory (Clemson University).

4.3. Experimental Plan and Preliminary Setup:

In order to precisely control the growth parameters during the process, we will enhance the sensing capabilities in our processing unit by employing thermocouples at various locations in the process tube and interface all the sensing and actuating devices to a mechatronics workstation operating under the real-time operating system QNX. Additional thermal sensing capabilities will also be integrated into to the

nanotube processing chamber to facilitate the development of differential equation based models for control synthesis.

The data acquisition and control implementation are performed at 500 kHz via standard I/O boards and standard interfacing circuitry via a breakout interface available on the nanotube processing chamber (see Fig. 5). The control algorithms are all implemented and run using the real-time control environment developed at Clemson University. This real-time control environment, QMotor 3.0, provides a versatile object-oriented framework for the implementation of advanced control algorithms as C++ programs. The QMotor 3.0 graphical user interface integrates functionality for testing and tuning of these control programs (e.g., data logging, plotting, and data exporting capabilities).

5. FUTURE RESEARCH PLAN AND APPROACH

As the next step under this task and through utilizing a type of coagulation-based carbon-nanotube spinning method [18, 23], we will fabricate piezoelectric polymer fibers. For this, we will inject CNTs into a variety bath of aqueous PVA to produce nanotube gel fibers, which will ultimately be washed to absorb the PVA and surfactant for use in the following tasks.

5.1. Development of CNT-based Mono- and Multi-filament Yarns:

Under this task, the fragile nanotubes gel fibers developed in the previous task will be pulled from coagulation bath at an optimum rate of about 1 cm/min (to be fine tuned later) and without removing PVA from the gel. It is expected that this method will produce gel fibers that are mechanically strong enough to be self-assembled into yarns (e.g., up to 10 cm in length) for a second spinning process. This stage of spinning process will involve unwinding the fibers onto a series of godets that carry them through an acetone-washing bath and then through a drying path so that they can be wrapped onto a mandrel. Unlike silkworm cocoons, which are made of single, continuous strands of silk fiber, the proposed nanotube-based arrays contain millions of separate CNTs. The thickness of the yarn depends on the size of the tip of the tool used to pull the nanotubes. It is expected that heating the yarn may strengthen the junctions between nanotubes (notice the ordinary weak van der Waals interaction force is relatively strong between nanotubes due to clean surface of the nanotube) which will ultimately increase yarn's tensile strength and conductivity [16]. The development of such continuous yarns made out of nanotubes would enable creation of macroscopic nanotube materials with desirable mechanical and electrical properties that can be eventually converted into fabrics for end use applications.

5.2. Design, Development and Control of Functional Fabrics with Actuation/Sensing Capabilities:

The unique electrical and mechanical properties of the long strands of CNTs (developed under previous task) would extend the remarkable properties of CNTs to a larger scale for weaving into a variety of macroscopic objects for different applications (just like silk thread in textile industry). As schematically shown in Fig. 6, the proposed e-textile will consist of composite fabric layers as distributed actuator and sensor segments. Each segment is comprised of woven CNT-

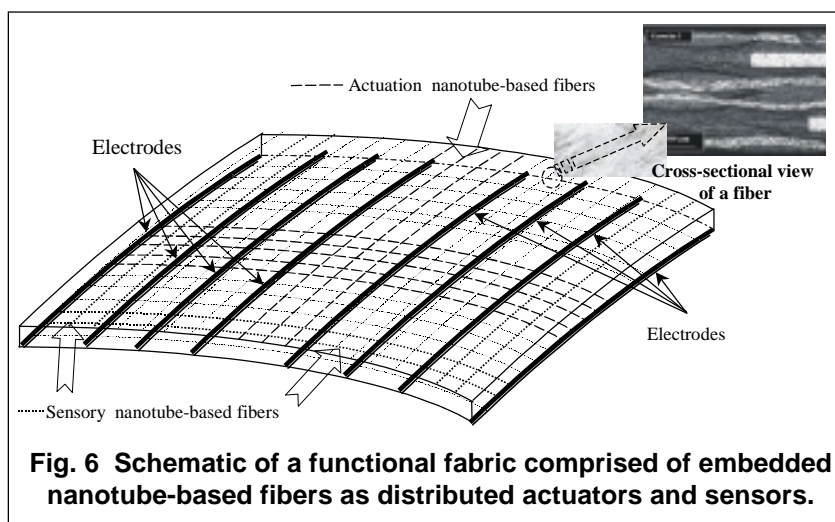
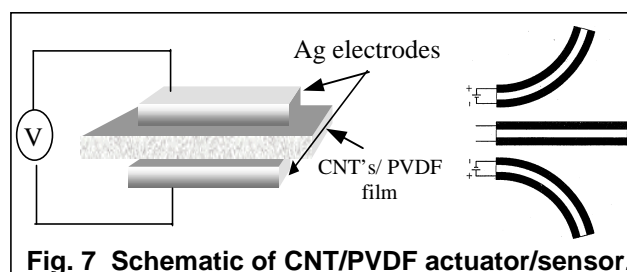


Fig. 6 Schematic of a functional fabric comprised of embedded nanotube-based fibers as distributed actuators and sensors.

based filament yarns sandwiched between two electrodes (see the detailed arrangement in Fig. 7). Preliminary data obtained in our laboratory for thin films of PVDF/CNT composites indicate that the addition of carbon nanotubes to the matrix gives an improved electromechanical response at lower voltages as compared to pure PVDF actuators/sensors [22].

The direct and reverse conversion of electrical energy into mechanical energy through nanotube-based actuators and sensors embedded into the fabric will create a platform for developing next-generation smart functional fabric. The sensing capabilities of the e-fabric will provide displacement measurements that can be used to represent the fabric shape and configuration. In response to these data, the actuators embedded in the fabric will be utilized to alter the shape of fabric to adapt to desired conditions (e.g., flying conditions in the case of aerostat aircraft). To this end, we will initially concentrate on the design and fabrication of smart fabric prototypes that can be used as building blocks for understanding the actuation, sensing and adaptation in response to external disturbances.



ACKNOWLEDGEMENT

We would like to thank the assistance of current graduate students both from the Mechanical Engineering Department (Mr. Laxminarayana, Mr. Ramaratnam, Mr. Rajoria and Mr. Cherian) and from the Electrical and Computer Engineering Department (Mr. Lonkar and Mr. Zhang). The financial support through National Textile Center (grant # M03-CL07s) is also highly appreciated.

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