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Development and characterisation of polyaniline - carbon nanotube conducting composite fibres

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**DEVELOPMENT AND CHARACTERISATION OF
POLYANILINE - CARBON NANOTUBE CONDUCTING
COMPOSITE FIBRES**

A thesis submitted in fulfillment of the
requirements for the award of the degree

DOCTOR OF PHILOSOPHY

From

UNIVERSITY OF WOLLONGONG



by

VAHID MOTTAGHITALAB

(B.Sc. Chemical Engineering; M.Sc. Polymer Engineering)

School of Material, Mechanical and Mechatronic Engineering

JUNE 2006

IN THE NAME OF ALLAH, THE MOST GRACIOUS, THE MOST MERCIFUL

This thesis is dedicated to:

My wife: Mahin

My son: Amir Reza

*For their love, support and patience
and my Mother for her encouragement,
especially in memory of my father
and*

To My Brothers

THESIS CERTIFICATION

I, Vahid Mottaghitalab, declare that this thesis, submitted in fulfillment of the requirements for the award of Doctor of Philosophy, in the School of Material, Mechanical and Mechatronic Engineering, Faculty of Engineering, University of Wollongong, is wholly my own work unless otherwise referenced or acknowledged. The thesis was completed under the supervision of Prof. Geoffery. M. Spinks and Prof. Gordon. G. Wallace and has not been submitted for qualification at any other academic institution.

Vahid Mottaghitalab

JUNE 2006

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PUBLICATIONS

Published Paper:

1. **V. Mottaghitalab**, G. M. Spinks and G. G. Wallace, The influence of carbon nanotubes on mechanical and electrical properties of polyaniline fibres, Synthetic Metals, 152, **(2005)**, 77-80.
2. G. M. Spinks, **V. Mottaghitalab**, M. Bahrami-Samani, P. G. Whitten and G. G. Wallace, Carbon Nanotube Reinforced Polyaniline Composite Fibres for High Strength Artificial Muscles, Advanced Materials, 18, (2006), 637.

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1. **V. Mottaghitalab**, B. Xi, G. M. Spinks and G. G. Wallace, Polyaniline Fibres Containing Single Walled Carbon Nanotubes: Enhanced Performance Artificial Muscles, Synthetic. Metals. **(2006)**.
2. **V. Mottaghitalab**, G. M. Spinks and G. G. Wallace, The development and characterization of polyaniline - single walled nanotube composite fibres using 2-acrylamido-2 methyl -1-propane sulfonic acid (AMPSA) through one step wet spinning process, Polymer, **(2006)**.

Submitted to Journals:

1. C. Y. Wang, **V. Mottaghitalab**, C. O. Too, G. M. Spinks and G. G. Wallace, Investigation of Polyaniline fibre as battery material in ionic liquid, Journal of power source, **(2006)**.

3. B. Xi, V.T. Truong, **V. Mottaghitalab**, P. Whitten, G. M. Spinks, G. G. Wallace, “ Actuation behavior of polyaniline films and tubes prepared by the phase inversion technique”, Submitted , Smart Mater. and Struct.(2005)

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1. **V. Mottaghitalab**, B. Xi, G. M. Spinks and G. G. Wallace, “The influence of carbon nanotubes on mechanical, electrical and electrochemical properties of polyaniline fibre”, ICSM, Wollongong, Australia, **2004**, Session: Electronic Fibres and Other Unconventional Substrates.
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4. G. M. Spinks, **V. Mottaghitalab**, Binbin Xi, P. Whitten, G. Wallace, S. J. Kim, S. R. Shin and S. I. Kim, Carbon Nanotube Reinforcement of Conducting Polymers and Hydrogels for High Strength Actuators, MRS Fall Meeting, W6.2/V7.2, Boston , MA ,USA, **(2005)**.
5. G. Spinks, B. Xi, T. Campbell, P. Whitten, **V. Mottaghitalab**, M. Bahrami Samani and G. G. Wallace, In pursuit of high-force/high-stroke conducting polymer actuators (Invited Paper), Proceedings of SPIE-The International Society for Optical Engineering, 5759,(2005), 314-321.

6. G. G. Wallace, **V. Mottaghitalab**, S. E. Moulton and a. G. M. Spinks., Synthesis and properties of organic, electronically conducting fibres: a platform for electronic textiles, Fr C3.2, Queenstown, New Zealand, **(2005)**.
8. B. Xi, V.-T. Truong, **V. Mottaghitalab**, P. Whitten, G. M. Spinks and G. G. Wallace, Ed: S. F. Al-Sarawi, Actuation behaviour of polyaniline films and tubes prepared by phase inversion technique, 5649, SPIE, Sydney, Australia, **(2005)**.436-444.

Manuscript in preparation:

1. **Vahid Mottaghitalab**, Philip G. Whitten, Geoffrey M. Spinks and Gordon G. Wallace , Nanotube orientation and load transfer in Polyaniline /Single Walled Carbon Nanotube Composite Fibre
2. **V. Mottaghitalab**, B. Xi, G. M. Spinks and G. G. Wallace, The study of electrical behavior of PANi- AMPSA fibre filled with CNTs: the enhanced metallic transport,

ABSTRACT

The present study describes methods for development and characterization of conducting electroactive polymer (CEP) fibre consisting of polyaniline (PAni) and single walled carbon nanotubes (SWNTs) which have potential applications as electronic devices to form building blocks of electronic textiles. The conducting composite fibres of PAni-SWNT were developed respectively using two steps (acid doping after fibre spinning) and one step methods (doping during preparation of spinning solution). The effectiveness of nanotube inclusion for improvement of mechanical, electrical and electrochemical properties was studied in each method. During development of the fibres, techniques such as UV-Vis-NIR, Raman spectroscopy, Dynamic light scattering and viscometry were used to characterise the quality of dispersion and spinning solutions. It has been shown that the N,N'-dimethyl propylene urea (DMPU) and dichloro acetic acid (DCAA) as solvents respectively for PAni in base and salt form are able to effectively disperse the pristine SWNTs to reach percolation level. The addition of nanotubes changes the rheological behavior of neat PAni spinning solution from a Newtonian to non-Newtonian shear thinning fluid based on power law regime which reflects nanotube-nanotube and/or nanotube-polymer physical entanglement. Several techniques including DMA, DSC, SEM, TEM, FIB, Raman spectroscopy, CV and four point probe electrical conductivity measurement were employed to characterize the various properties of the solid fibre. In both, one step or two steps methods, fibres containing SWNTs have superior tensile strength and elastic modulus compared with neat PAni fibre. The inclusion of SWNTs to PAni, however, decreases the elongation at break. These outcomes directly can be

attributed to physical and/or chemical interfacial interaction between well distributed SWNTs bundles and the PANi matrix. The addition of nanotubes to the PANi matrix also increases the electrical conductivity and enhances the electrochemical redox process. However, the two step method was found to have some problem include low spinning rate, low flexibility and low conductivity and insufficient charge transfer along the fibre to be working electrode. These disadvantages were diminished by faster spinning of PANi-ES/2-acrylamido-2 methyl -1-propane sulfonic acid (AMPSA)/SWNT using the one step process with more than 5 times stretching ratio. An electronic conductivity percolation threshold of ~ 0.35 % w/w SWNTs was determined with fibres possessing electronic conductivity up to $\sim 750 \text{ Scm}^{-1}$. The well defined electrochemical window for neat PANi-ES/AMPSA fibre and its composite containing SWNT either in aqueous or ionic liquid electrolyte, with wider electrochemical window, confirms the ease of charge transport through a new conduction path for the fibre formed from salt structure, which was enhanced by addition of nanotubes. The ultimate tensile strength, elastic modulus and elongation at break of PANi-ES/AMPSA/SWNT fibres containing 0.76 % w/w nanotubes respectively were obtained $255 \pm 32 \text{ Mpa}$, $7.3 \pm 0.4 \text{ GPa}$ and 4 ± 1 % compared with $170 \pm 22 \text{ MPa}$, $3.4 \pm 0.4 \text{ GPa}$ and 9 ± 3 % for PANi-ES/AMPSA fibre. The quantitative analysis of nanotube orientation and detection of load transfer from matrix to nanotubes were investigated in PANi-ES/AMPSA/SWNT composite fibre using Raman spectroscopy. It has been found that thermal stretching of as spun fibre mostly orients the nanotubes in a range of about $\pm 30^\circ$ versus fibre axis which extremely increase the Herman orientation factor from 0.02 for as spun fibre to 0.43 for the 5x drawn fibre. Moderate orientation and Raman shift about $90\text{-}130 \text{ cm}^{-1}$ in D^* band of SWNT also can

be correlated to effective but not perfect load transfer between PANi matrix and nanotubes. The result of temperature dependent electrical conductivity data was shown that the higher conductivity of PANi-ES/AMPSA/SWNT composite fibre compared to neat PANi-ES/AMPSA fibre also can be described by improvement of the metallic property in the crystalline areas and boosting of the metallic disorder contribution in amorphous area. The consequence of improvement of mechanical, electrical and electrochemical properties were a benefit for applying of PANi/ES-AMPSA fibre and its composite having SWNT in applications as actuator, power source and sensor. While the fibres showed great promise as actuators, their response as batteries and temperature/humidity sensors was limited. The significant improvement was observed in actuator strength in excess of 100 MPa and work-per-cycle of over 300 kJ/m³ through the incorporation of small amounts of SWNTs as reinforcement in the PANi matrix. This performance is 3 times higher than previously produced conducting polymer actuators and exceeds skeletal muscle in terms of stress generation by 300 times. PANi-ES/AMPSA/SWNT exhibited a higher charge/discharge capacity (12.4/11.2 mAhg⁻¹) compared with the neat PANi-ES/AMPSA (4.5/4.1 mAhg⁻¹). All the results show that solid polyaniline fibre can be used directly as electrode in ionic liquid of EMI.TFSI for wearable power source system. However its current performance is still well below conventional rechargeable battery systems. PANi fibre and its SWNT composite showed a nonlinear response with some delay to temperature signals. The PANi fibre incorporated with SWNTs showed lower sensitivity to change in humidity pulse compared with neat PANi fibre. This behavior has good opportunity for application in conducting yarn that

needs the lowest variability in conductivity for transferring of electrical signal but clearly is not favored for sensing of humidity.

Abbreviations

Ch	Chiral vector
MWNT	multi wall carbon nanotubes
n	Power law index
NIBS	Non invasive backscattering
NIR	near infra red
NMP	N- methyl pyrrolidinone
n_R	The solvent refractive index
Nylon	11 - poly ω -aminoundecanoyl
ODF	Orientation distribution function
PAN	Polyacrylonitrile
PAni	Polyaniline
PBO	Poly butyl oxide
PGB	Pernigraniline base
Pi (cos θ)	The Legendre polynomial of degree i
$\langle P_2(\cos\theta) \rangle$	The average amount of $P_2(\cos\theta)$
$\langle P_4(\cos\theta) \rangle$	The average amount of $P_4(\cos\theta)$
POT	polyoctylthiophene
PPMS	Physical property measurement system
PPy	Polypyrrole
Psi	Pound per square inch
PT	Polythiophene
PVA	Poly vinyl alcohol
Q	volumetric flow rate (cm ³ /min)
R	Resistance(ohm)
RBM	Resonance breathing mode
S	Cross sectional area of fibre (cm ²)
SEM	Scanning electron microscopy

SWNT	single wall carbon nanotubes
$S\sigma$	the critical exponent at percolation
T	Temperature (K, °C)
TEM	Transmission electron microscopy
T _g	glass transition temperature (°C)
TGA	Thermogravimetric analysis
TM	Tangential mode (G band)
TPa	Tera pascal
T _t and T _s	tunneling parameters for transport model(K)
V	Voltage (V, mV)
V _d	Drawing velocity (m/min)
V _i	Injection rate (g/min)
V _t	Take up velocity (m/min)
α	Chiral angle
γ	Shear rate (s ⁻¹)
δ_b	Tensile strength at break (MPa)
$\epsilon_{c,m,f}$	Expected actuator strain of composite, matrix or filler
ϵ_b	Elongation at break (%)
η	Viscosity (mPa.s=cP)
η_0	Zero shear viscosity (mPa.s=cP)
θ	The scattering angle of laser beam
θ°	The angle between nanotubes and fibre axis
λ_2 and λ_4	The arbitrary parameter of orientation function
λ_o	The vacuum wavelength of the laser
ρ	Density (g/cm ³)
ρ_i	Resistivity of a potion either amorphous or crystalline
σ	Conductivity (S/cm)
ϕ	Spin draw ratio
ϕ_{th}	Thermal draw ratio

ψ	The angle between the polarization plane and the fibre axis
v_i	Volume fraction of component i
ω	SWNTs mass fraction in the solid fibre
ω_1	The contribution of metallic resistivity in bulk resistivity
ω_2	The contribution of nonmetallic resistivity in bulk resistivity
ω_c	critical weight percentage of SWNT at percolation
ω_p	weight fraction of polymer with regards to solvents

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