

STRETCHABLE AND FLEXIBLE CONDUCTIVE FIBERS

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STRETCHABLE AND FLEXIBLE CONDUCTIVE FIBERS

by

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Dedicated to My Parents, Prof. Manjeet Jassal &

Prof. Ashwini K. Agrawal

*You showed me light when I was in dark
In my life you have left a very big mark
In every step, you were my guide
My every success is your pride*

CERTIFICATE

This is to certify that the thesis titled ‘**Stretchable and Flexible Conductive Fibers**’, being submitted by **Ms. Kiran Yadav** to the **Indian Institute of Technology Delhi** for the award of degree **Doctor of Philosophy**, is a record of bonafide research work carried out by her. She has worked under my guidance and supervision and fulfilled the requirements for the submission of thesis, which has attained the standard required for a PhD. degree of this Institute.

The results contained in this thesis have not been submitted, in part or in full, to any other university or institute for the award of any degree or diploma.

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ABSTRACT

Technological advancement in the area of electronics demands light, bendable, foldable, conformable and portable devices. In order to meet these requirements, the development is moving towards flexible and stretchable solutions. Literature reports various conducting nanomaterials for creating the conductive paths in film and fiber configuration. Fibers are mechanically stronger and more flexible compared to films for their use in various applications and they could also be integrated into textiles.

Silver nanowires (AgNWs) are a promising candidate for making flexible and stretchable electronics due to the highest electrical conductivity of silver in its bulk state. In the present study, synthesis of AgNWs using template-less approaches has been studied. AgNWs with aspect ratio of ~ 164 having diameter of 55 ± 9 nm were synthesized following glycerol mediated solvothermal method. The dispersion behavior of isolated and wet AgNWs in different solvents namely, ethanol, acetone and dimethylformamide was also investigated.

The incorporation of wet AgNWs in electrospun fibers through electrospinning was explored. PVA nanofibers having uniformly distributed, well aligned, and highly inter-connected AgNWs were electrospun. Using this approach, a very high amount of nanowires (up to 34.5 wt%) could be successfully incorporated into the nanofibers. The incorporation and alignment of AgNWs was attributed to the interaction of AgNWs with PVA and the polarization of long nanowires during the electrospinning process. A free standing, highly conducting nanoweb was fabricated with conductivity of approximately 654.85 S/cm. These conductive nanowebs are of interest for a number of electronic devices such as sensors or energy storage systems requiring flexibility and high surface area.

A new approach for making stable, flexible and conductive hollow fibers using dry-jet-wet spinning technique was investigated using poly(acrylonitrile) (PAN) as the polymer. The inner surface of the AgNW-PAN hollow fibers was deposited with AgNWs using its dispersion in the bore fluid. The bore fluid was found to play a crucial role in determining the morphology and flexibility of the hollow fibers and entrapment of long AgNWs on the inner walls. Highly conducting fibers with conductivity of $\sim 10^4$ S/cm could be obtained with the use of ~ 2 wt% of AgNWs. Using the above approach, highly stretchable, flexible conducting hollow fibers were also solution spun using intrinsically stretchable polyurethane. The influence of AgNWs concentration and bore fluid flow rate on the conductivity and stretchability of the hollow fibers was investigated. These fibers showed very high conductivity of $\sim 1.52 \times 10^4$ S/cm at a very low concentration of AgNWs dispersion (~ 0.46 wt%). They also showed good retention of conductivity even at 100% strain, which remained stable with repeated cycling. These properties were mainly attributed to the deposition of AgNWs in the form of three distinct layers, namely embedded, anchoring and topmost AgNWs clusters.

Finally, the feasibility of the spun conductive hollow fibers as an electrode material for flexible supercapacitor was explored. The conducting fibers were successfully assembled into coaxial configuration to yield highly stable, flexible supercapacitors with capacitance value of 128 F/cm^3 . The unique morphology of these conductive hollow fibers opens the possibility of making flexible and stable devices for wearable electronics.

The work discussed in this dissertation conclusively showed the significance of the preparation of desirable nanomaterials and the design of effective nanostructured electrodes for supercapacitor energy storage applications.

सार

इलेक्ट्रॉनिक्स के क्षेत्र में तकनीकी उन्नति, हलके, वक्रीय, आकृति अनुरूप और वहनीय उपकरणों की मांग करती है। इन आवश्यकताओं को पूरा करने के लिए, विकास लचीले और खींचने योग्य समाधान की ओर बढ़ रहा है। फिल्म और फाइबर कॉन्फिगरेशन में प्रवाहकीय पथ बनाने के लिए साहित्य विभिन्न नैनो-मटेरियल्स की रिपोर्ट करता है। रेशे, फिल्मों की तुलना में यंत्रवत् रूप से मजबूत और अधिक लचीले होने के कारण विभिन्न अनुप्रयोगों में उपयोग किए जाते हैं तथा वे वस्त्रों में एकीकृत भी हो सकते हैं। रजत नैनोवायर्स (AgNWs) अपने थोक अवस्था में चांदी के उच्चतम विद्युत चालकता के कारण लचीले और खिंचावयोग्य इलेक्ट्रॉनिक्स बनाने के लिए एक आशाजनक उम्मीदवार हैं। वर्तमान अध्ययन में, टेम्पलेट-मुक्त दृष्टिकोण का उपयोग करते हुए AgNWs के संश्लेषण का अध्ययन किया गया है। ग्लिसरॉल मध्यस्थता वाले सॉल्वैथर्मल विधि के अनुसार 55 ± 9 nm के व्यास वाले ~ 164 के पहलू अनुपात के साथ AgNWs संश्लेषित किए गए हैं। अलग-अलग सॉल्वेंट्स जैसे इथेनॉल, एसीटोन और डाइमिथाइल फॉर्ममाइड (DMF) में पृथक एवं गीले AgNWs के फैलाव व्यवहार की जांच भी की गई।

इलेक्ट्रोस्पिनिंग के माध्यम से इलेक्ट्रोस्पिन रेशे में गीले AgNWs के समावेश का पता लगाया गया है। AgNWs को PVA के अतिसूक्ष्म रेशो में समान रूप से वितरित, पंक्तिबद्ध और अत्यधिक अंतःपरस्पर जुड़े हुए पाया गया। इस दृष्टिकोण का उपयोग करते हुए, नैनोवायर की एक बहुत बड़ी मात्रा (34.5 wt%) को सफलतापूर्वक नैनोफाइबर्स में शामिल किया जा सका। AgNWs के निगमन और संरेखण को PVA के साथ AgNWs के संपर्क और इलेक्ट्रोस्पिनिंग प्रक्रिया के दौरान लंबे नैनोवायर्स के धुवीकरण का श्रेय दिया गया। एक स्वतंत्र, अत्यधिक संचालन लगभग 654.85 S/cm की चालकता के साथ नैनोवेब गढ़ा गया

था। ये प्रवाहकीय नैनोवेब कई ऐसे इलेक्ट्रॉनिक उपकरणों के लिए दिलचस्प हैं जिन प्रणालियों को लचीलेपन और उच्च सतह क्षेत्र की आवश्यकता होती है जैसे सेंसर या ऊर्जा भंडारण।

ड्राय-जेट-वेट कटाई तकनीक का उपयोग करके स्थिर, लचीले और प्रवाहकीय खोखले रेशे बनाने के लिए एक नए दृष्टिकोण के उपयोग से पाली (एक्रिलोनिट्राइल) (PAN) बहुलक की जांच की गई। AgNW-PAN खोखले फाइबर की आंतरिक सतह बोर द्रव में फैलाव का उपयोग करते हुए AgNWs के साथ जमा की गई थी। बोअर द्रव का खोखले फाइबर की आकृति विज्ञान, लचीलेपन और आंतरिक दीवारों पर लंबे समय तक AgNWs के फंसाने में महत्वपूर्ण भूमिका अवलोकित हुई। AgNWs के $\sim 2\%$ के प्रयोग से $\sim 10^4$ S/cm की चालकता के साथ उच्च चलित तंतुओं को प्राप्त किया जा सका। उपर्युक्त दृष्टिकोण का उपयोग, उच्च फैलने योग्य, आंतरिक रूप से खिंचाव वाले पॉलीयुरेथेन का इस्तेमाल करते हुए संचालन करने योग्य खोखले, फाइबर की संरचना के लिए किया गया। AgNWs की एकाग्रता और खोखले फाइबर के प्रवाहकत्व पर प्रवाह द्रव प्रवाह दर का प्रभाव जांच कर लिया गया। इन फाइबर में AgNW के फैलाव (~ 0.46 wt%) की बहुत कम एकाग्रता में $\sim 1.52 \times 10^4$ S/cm की बहुत अधिक चालकता दिखायी गयी। उन्होंने 100% तनाव पर भी चालकता का अच्छा प्रतिधारण दिखाया, जो साइकिलिंग के साथ स्थिर रहा। इन गुणों को मुख्य रूप से AgNW के तीन अलग-अलग परतों, अर्थात् एम्बेडेड, एंकरिंग और सबसे ऊपर के AgNWs क्लस्टर में जमा होने की वजह से पाया गया।

अंत में, लचीला सुपरकैपेसिटर के लिए इलेक्ट्रोड सामग्री के रूप में प्रवाहकीय खोखले फाइबर की व्यवहार्यता का पता लगाया गया। संचालन फाइबर को सफलतापूर्वक समाक्षीय कॉन्फिगरेशन में इकट्ठा किया गया था, जो 128 F/cm³ के समाई मूल्य के साथ अत्यधिक

स्थिर, लचीला सुपरकैपेसिटर उत्पन्न करते हैं। इन प्रवाहकीय खोखले फाइबर के अद्वितीय आकारिकी, पहनने योग्य इलेक्ट्रॉनिक्स के लिए लचीला और स्थिर उपकरण बनाने की संभावना को खोलते हैं।

इस निबंध में चर्चा की गई कार्यवाही ने वांछनीय नैनो-मटेरियल्स की तैयारी का महत्व और सुपर-कैपेसिटर ऊर्जा भंडारण अनुप्रयोगों के लिए प्रभावी नैनोस्ट्रक्चर्ड इलेक्ट्रोड की रचना को दिखाया।

TABLE OF CONTENTS

CONTENTS	Page No.
Certificate	i
Acknowledgements	iii
Abstract	v
Table of contents	vii
List of figures	xiii
List of tables	xxv
List of schemes	xxvii
List of symbols and abbreviations	xxix
Chapter 1: Introduction and literature review	
1.1 Introduction	1
1.2 Approaches for making stretchable and flexible electronics	1
1.2.1 Using intrinsically conducting polymers	2
1.2.2 Using carbon based conducting nanomaterials	2
1.2.3 Using metallic conducting nanomaterials	4
1.3 Synthesis of silver nanowires	5
1.3.1 Microwave assisted synthesis	5
1.3.2 Ultraviolet assisted synthesis	6
1.3.3 Arc discharge method	6
1.3.4 Hydrothermal assisted synthesis	6
1.3.5 Solvothermal method	7

1.4	Conductive polymer composites for flexible electronics	11
1.4.1	Conducting films	11
1.4.2	Conducting fibers	15
1.5	Conductive polymer composites applications	23
1.5.1	Strain sensors	23
1.5.2	Supercapacitors	23
1.5.3	Stretchable interconnects	24
1.5.4	Photovoltaics	25
1.6	Motivation	25
1.7	Objectives of the current work	27
1.8	Organization of thesis	28

Chapter 2: Synthesis and characterization of silver nanowires

2.1	Introduction	29
2.2	Experimental Methods	29
2.2.1	Materials	29
2.2.2	Synthesis of AgNWs using hydrothermal route	30
2.2.3	Synthesis of AgNWs using polyol mediated solvothermal route	32
2.2.4	Effect of process parameters on NaCl mediated synthesis	34
2.2.5	Dispersion of AgNWs	35
2.2.6	Characterization	35
2.3	Results and Discussion	36
2.3.1	Synthesis of AgNWs using hydrothermal route	36

2.3.2	Synthesis of AgNWs using polyol mediated solvothermal route	45
2.3.3	Dispersion behavior of AgNWs in solvents	63
Chapter 3: Conductive AgNW-PVA nanowebs		
3.1	Introduction	71
3.2	Experimental Section	71
3.2.1	Materials	71
3.2.2	Synthesis of AgNWs	72
3.2.3	Preparation of AgNW-PVA solutions	72
3.2.4	Electrospinning of PVA and AgNW-PVA solutions	72
3.2.5	Characterization	73
3.3	Results and Discussion	74
3.3.1	Morphology of PVA and AgNW-PVA nanofibers	74
3.3.2	Interaction between AgNWs and polymer PVA	79
3.3.3	Incorporation of AgNWs in PVA nanofibers	83
3.3.4	Flexible AgNW-PVA Nanoweb	85
3.3.5	Flexible Conductive AgNW-PVA Nanoweb	85
Chapter 4: Flexible and conductive AgNW-PAN hollow fibers		
4.1	Introduction	89
4.2	Experimental Section	89
4.2.1	Materials	89
4.2.2	Synthesis of AgNWs	90
4.2.3	Solution spinning of PAN and AgNW-PAN hollow fibers	90

4.2.4	Effect of AgNWs concentration in bore fluid and its flow rate on solution spinning of PAN hollow fibres	91
4.2.5	Characterization	92
4.3	Results and Discussion	92
4.3.1	Spinning of PAN hollow fibers	93
4.3.2	Spinning of flexible conducting hollow fiber	95
4.3.3	Effect of bore fluid flow rate	97
4.3.4	Effect of AgNW concentration in bore fluid	103
4.3.5	Flexibility testing	105
4.3.6	Conductive AgNW-PAN hollow fiber as electrical cable	107
4.3.7	Effect of ageing	109

Chapter 5: Highly stretchable, flexible and conductive AgNW-PU hollow fibers

5.1	Introduction	111
5.2	Experimental Methods	111
5.2.1	Materials	111
5.2.2	Synthesis of AgNWs	111
5.2.3	Fabrication of conductive hollow polyurethane fibers	112
5.2.4	Characterization	112
5.3	Results and Discussion	113
5.3.1	Morphology of AgNW-PU hollow fibers	113
5.3.2	Effect of bore fluid concentration	114
5.3.3	Effect of bore fluid flow rate	117

5.3.4	Flexibility testing	122
5.3.5	Stretchability testing	122
5.3.6	Ageing test	133
Chapter 6: Flexible supercapacitor using AgNW-Polymer hollow fibers		
6.1	Introduction	135
6.2	Experimental Section	135
6.2.1	Materials	135
6.2.2	Synthesis of AgNWs	135
6.2.3	Spinning of conductive PAN hollow fibers	136
6.2.4	Preparation of electrolyte solution	138
6.2.5	Fabrication of supercapacitor	138
6.2.4	Characterization	140
6.3	Results and Discussion	140
6.3.1	Electrochemical analysis of parallel assembly	140
6.3.2	Electrochemical analysis of co-axial assembly	143
6.3.3	Flexibility testing of coaxial supercapacitor	147
6.3.4	Cyclic testing of coaxial supercapacitor	148
Chapter 7: Conclusions		151
References		155
Appendix		167
Brief Bio-data of the author		171

LIST OF FIGURES

Figure No.	Figure caption	Page No.
Figure 2.1	SEM micrographs of silver nanostructures obtained through hydrothermal route, using PVP as the reducing agent, at two different positions and magnifications of (a) 3000 and (b) 24000×	37
Figure 2.2	Silver nanostructures obtained through hydrothermal route, using PVP as the reducing agent and reduced NaCl concentration (a) Shiny layer formed on the surface of as synthesized dispersion (b) SEM micrograph of the unwashed silver nanostructures at 5000×	38
Figure 2.3	SEM micrographs of silver nanostructures obtained through hydrothermal route using D-glucose as the reducing agent, at magnifications of (a) 2500 and (b) 5000×	39
Figure 2.4	SEM micrographs of AgNWs synthesized by hydrothermal route via interfacial chemistry at magnifications of (a) 2400 and (b) 10000×	40
Figure 2.5	(a) Electron image and (b) Elemental mapping of AgNWs synthesized by hydrothermal route via interfacial chemistry	41
Figure 2.6	EDX analysis of AgNWs synthesized by hydrothermal route via interfacial chemistry	42
Figure 2.7	SEM micrographs of AgNWs synthesized by hydrothermal route via interfacial chemistry, with two times increase in batch size, at magnifications of (a) 10000 and (b) 20000×	43

Figure 2.8	SEM micrographs of AgNWs synthesized by hydrothermal route via interfacial chemistry, with three times increase in batch size using 3 M AgNO ₃ solution, at two different places (a) and (b) with magnification of 6000×	43
Figure 2.9	SEM micrographs of AgNWs synthesized by hydrothermal route via interfacial chemistry, with three times increase in batch size using 1 M AgNO ₃ solution, at magnifications of (a) 5000 and (b) 12000×	44
Figure 2.10	SEM micrograph of Ag nanostructures synthesized by ethylene glycol mediated solvothermal route, at two different positions and magnifications of (a) 10000 and (b) 40000×	45
Figure 2.11	SEM images of Ag nanostructures synthesized via CuCl ₂ .2H ₂ O mediated ethylene glycol based solvothermal route, at two different positions and magnifications of (a) 200 and (b) 10000×	46
Figure 2.12	SEM micrographs of Ag nanostructures synthesized via NaCl mediated ethylene glycol based solvothermal route using PVP K-90, at magnifications of (a) 1000 and (b) 10000×	47
Figure 2.13	SEM micrographs of Ag nanostructures synthesized via NaCl mediated ethylene glycol based solvothermal route using PVP-K30, at magnifications of (a) 2000 and (b) 13000×	48
Figure 2.14	SEM images of AgNWs synthesized via NaCl mediated ethylene glycol based solvothermal route using PVP-K30 with 5 fold increase in the volume, at magnifications of (a) 1000 and (b) 15000×	49

Figure 2.15	SEM micrographs of AgNWs synthesized via NaCl mediated ethylene glycol based solvothermal route using PVP-K30 with 10 fold increase in the volume, at magnifications of (a) 5000 and (b) 10000×	50
Figure 2.16	SEM micrographs of AgNWs synthesized via NaCl mediated ethylene glycol based solvothermal route using PVP-K30 with 5 fold increase in the volume and two fold increase in the concentrations, at magnifications of (a) 1500 and (b) 5000×	50
Figure 2.17	SEM micrographs of AgNWs synthesized via NaCl mediated ethylene glycol based solvothermal route using PVP-K30 with five fold increase in the volume and four fold in the concentrations, at magnifications of (a) 5000 and (b) 10000×	51
Figure 2.18	SEM images of Ag nanostructures obtained on decreasing reaction temperature from 170 to 150 °C, at magnifications of (a) 2400 and (b) 5000×	52
Figure 2.19	SEM images of silver nanocubes obtained on decreasing the injection rate of precursor solution, at two different places (a) and (b) at magnification of 5000×	53
Figure 2.20	SEM images of silver nanoparticles obtained on decreasing the molar ratio of PVP: AgNO ₃ from 7.5 to 5, at two different places (a) and (b) at magnification of 5000×	54
Figure 2.21	SEM images of silver nanoparticles obtained on decreasing the molar ratio of AgNO ₃ : NaCl, at magnifications of (a) 3000 and (b) 6000×	54

Figure 2.22	SEM micrographs of silver nanostructures present in three layers of as synthesized dispersion, (a) upper layer, (b) middle layer and (c) bottom layer at 40000×	57
Figure 2.23	SEM micrographs of AgNWs obtained through glycerol mediated polyol synthesis, at magnifications of (a) 5000 and (b) 160000× and (c) TEM micrograph of AgNWs showing diameter of 49 nm	58
Figure 2.24	EDX micrograph of AgNWs obtained through glycerol mediated polyol synthesis, (a) electron image and (b) Ag L α 1 elemental image	59
Figure 2.25	(a) AFM scan and (b) the histogram showing the height/diameter of the AgNWs obtained through glycerol mediated solvothermal synthesis	60
Figure 2.26	Conductivity testing of AgNWs synthesized via glycerol mediated solvothermal method using LED in the circuit	61
Figure 2.27	UV-visible spectrum of AgNWs synthesized via glycerol mediated solvothermal method dispersed in DI water	62
Figure 2.28	Raman spectrum of isolated dried AgNWs	63
Figure 2.29	Photograph of the dispersions of AgNWs (isolated dried) in different solvents	64
Figure 2.30	SEM micrographs showing the effect of solvent system on dispersion of isolated AgNWs in (a) ethanol, (b) DMF and (c) acetone	65

Figure 2.31	SEM micrograph showing the effect of sonication time on dispersion of 0.05 wt% isolated AgNWs in (a-c) ethanol and (d-f) DMF for 5, 15 and 30 min, respectively	66
Figure 2.32	SEM micrographs of isolated dried AgNWs at magnifications of (a) 400 and (b) 10000×	67
Figure 2.33	SEM micrographs of (a) as obtained and (b) diluted wet AgNWs	68
Figure 2.34	SEM micrographs showing the effect of solvent system on dispersion of wet AgNWs in (a) ethanol, (b) DMF and (c) acetone	69
Figure 3.1	SEM micrographs of PVA and AgNW-PVA composite nanofibers with 5.75-34.5 wt% AgNWs at 40000× magnification	75
Figure 3.2	Elemental carbon and silver mapping of AgNW-PVA composite nanofibers having 5.75 and 11.5wt% of AgNWs	77
Figure 3.3	Elemental carbon and silver mapping of AgNW-PVA composite nanofibers having 17.25 and 34.5 wt% of AgNWs	78
Figure 3.4	Raman spectra from 100-300 cm ⁻¹ for (a) PVA solution, (b) AgNW aqueous dispersion and (c) AgNW/PVA dispersion	79
Figure 3.5	Schematic showing interaction mechanism of AgNWs with PVP and PVA	80

Figure 3.6	Raman spectra: 500-800 cm^{-1} for (a) PVA solution, (b) AgNW aqueous dispersion and (c) AgNW/PVA dispersion	80
Figure 3.7	Raman spectra: 1500-1800 cm^{-1} for (a) PVA solution, (b) AgNWs aqueous dispersion and (c) AgNW/PVA dispersion	81
Figure 3.8	Raman spectra: 100-3200 cm^{-1} for (a) PVA nanoweb, (b) AgNW-PVA film and (c) AgNW-PVA nanoweb	82
Figure 3.9	Flexible free standing 34.5AgNW-PVA composite nanoweb	85
Figure 3.10	Photographic image of (i) as-spun 34.5AgNW-PVA nanoweb (ii) treated 34.5AgNW-PVA nanoweb, placed in series with LED circuit	86
Figure 3.11	Resistance measurement of 34.5AgNW-PVA nanoweb using multimeter	86
Figure 3.12	SEM micrographs of partially exposed 34.5AgNW-PVA electrospun nanoweb at magnifications of a) 10000 and b) 20000 $\times$	87
Figure 4.1	Representative dry-jet-wet spun PAN hollow fiber	93
Figure 4.2	SEM micrographs of hollow PAN fibers spun at BFFR of (a) 10, (b) 20 and (c) 40 ml/h	94
Figure 4.3	SEM micrograph of hollow fiber spun using aqueous bore fluid at BFFR of 40 ml/h	95

Figure 4.4	SEM micrograph of hollow fiber spun using 0.1wt% AgNW (water:DMF) dispersion in 50:50 vol/vol as bore fluid at BFFR of 40 ml/h	96
Figure 4.5	SEM micrographs of cross-section of AgNW-PAN hollow fibers spun at different BFFR using 0.1 wt% AgNW (water: DMF::50:50) dispersion	98
Figure 4.6	SEM micrographs of longitudinal section of AgNW-PAN hollow fibers spun at different BFFR using 0.1 wt% AgNW (water: DMF::50:50) dispersion	99
Figure 4.7	Current vs. voltage profile of 5 cm long AgNW-PAN hollow fibers spun at different BFFR using 0.1 wt% AgNW (water: DMF::50:50) dispersion	101
Figure 4.8	Conductance of AgNW-PAN hollow fibers spun at different BFFR using 0.1 wt% AgNW (water:DMF::50:50)	102
Figure 4.9	SEM micrographs of longitudinal section of AgNW-PAN hollow fibers spun at 60 ml/h with varying AgNW concentration	103
Figure 4.10	Current vs. voltage profile of 5 cm long AgNW-PAN hollow fibers	104
Figure 4.11	Effect of AgNWs concentration on the conductance of AgNW-PAN hollow fibers	105
Figure 4.12	Photographs showing wrapping of 0.1NW-FR100 hollow fiber over 2.5 mm radius rod	106

Figure 4.13	Effect of knot on the current vs. voltage profile of AgNW-PAN conductive hollow fiber	106
Figure 4.14	Change in resistance of 0.1NW-FR80 conductive hollow fiber at 5% strain for 50 cycles	107
Figure 4.15	AgNW-PAN conductive hollow fiber in-line with LED working as an electrical wire	108
Figure 4.16	Effect of ageing on current passing capacity of AgNW-PAN conductive hollow fiber	108
Figure 5.1	SEM micrographs of (a) AgNW-PU hollow fiber cross-section at 200 $\times$, (b) magnified view of AgNWs adhered to the fiber's inner surface at 80000 $\times$	114
Figure 5.2	SEM micrographs of longitudinal section of AgNW-PU hollow fibers spun at BFFR of 20 ml/h with varying AgNWs concentrations (a) 0.05, (b) 0.08, (c) 0.1, and (d) 0.2 wt%	115
Figure 5.3	Current vs. voltage profile for 5 cm long AgNW-PU hollow fibers	116
Figure 5.4	Effect of AgNW concentration on the conductance of AgNW-PU hollow fibers	117
Figure 5.5	SEM micrographs of cross-section of AgNW-PU hollow fibers spun at different BFFR using 0.2 wt% AgNWs dispersion	118
Figure 5.6	Current vs. voltage profile for conductive 5 cm long hollow fibers spun at different BFFR	119

Figure 5.7	Conductance of conductive hollow fibers spun at different BFFR	120
Figure 5.8	Effect of knot on the current-voltage profile of 0.2NW-FR60 fiber	122
Figure 5.9	Effect of strain on the conductance of 5 cm long hollow fibers with different concentrations of AgNWs	123
Figure 5.10	Change in resistance of 0.2NW-FR60 conductive hollow fiber at 20% strain for 70 cycles	124
Figure 5.11	SEM micrographs representing the inner surface of 0.2NW-FR60 conductive hollow fiber (a) & (b) at 20% and (c) & (d) at 0% strains after 70 cycles	125
Figure 5.12	Change in resistance of 0.2NW-FR60 conductive hollow fiber at 20 and 100% strain for 70 cycles	126
Figure 5.13	SEM micrographs representing the inner surface of 0.2NW-FR60 conductive hollow fiber (a) & (b) at 100% and (c) & (d) at 0% strains after 70 cycles	127
Figure 5.14	Schematic representation of conductive hollow fiber longitudinal section along with SEM micrographs representing (a) top surface, (b) the anchoring layer, (c) the embedded layer of 0.2NW-FR60 at different positions at 100% stretched condition after 70 cycles	129
Figure 5.15	Change in resistance of 0.1NW-FR60 conductive hollow fiber at 100% for 70 cycles in comparison with 0.2NW-FR60	131

Figure 5.16	SEM micrographs representing the inner surface of 0.1NW-FR60 conductive hollow fiber (a) & (b) at 100% and (c) & (d) at 0% strains after 70 cycles	132
Figure 5.17	LED connected in-line with 0.2NW-FR60 conductive hollow fiber, at 0 & 100% strain	133
Figure 5.18	Effect of ageing on the current passing capacity of hollow fiber at 3 V	134
Figure 6.1	Cyclic voltagrams of parallel assembled AgNW-PAN conductive hollow fibers spun at different BFFR	141
Figure 6.2	Effect of voltage scan rate on the volumetric capacitance of 0.1NW-FR120/120 supercapacitor assembly	142
Figure 6.3	Cyclic voltagrams of coaxially assembled fibers 0.1NW-FR120 and 0.1NW-FR40 having only annulus filled, and both core and annulus filled geometry	143
Figure 6.4	Effect of voltage scan rate on the volumetric capacitance for core and annulus filled 0.1NW-FR120/40 and annulus filled 0.1NW-FR120/40 supercapacitor fiber assemblies	144
Figure 6.5	Nyquist plot showing electrochemical impedance of core and annulus filled 0.1NW-FR120/40 and annulus filled 0.1NW-FR120/40 supercapacitor assemblies	145
Figure 6.6	Galvanostatic charge discharge curves of coaxially assembled fibers 0.1NW-FR120 and 0.1NW-FR40 having only annulus filled, and both core and annulus filled geometry at 20 μ A	146

Figure 6.7	Cyclic voltagrams of core and annulus filled supercapacitor assembly in straight and looped condition with a photograph showing fabricated supercapacitor with a loop of 360°	148
Figure 6.8	Cyclic voltagram of core and annulus filled supercapacitor assembly for 35 cycles at 200 mV/s	149
Figure 6.9	Percentage retention in volumetric capacitance of CA-0.1NW-FR120/40 assembly for 35 cycles	149

LIST OF TABLES

Table No.	Title of the Table	Page No.
Table 1.1	Flexible and stretchable conductive films using vacuum filtration approach	12
Table 1.2	Flexible and stretchable conductive films using coating technique	13
Table 1.3	Flexible and stretchable conductive films using in-situ polymerization	14
Table 4.1	Dimensional and electrical properties of AgNW-PAN hollow fibers spun at different BFFR	100
Table 5.1	Structural parameters and electrical properties of conductive hollow fibers spun at different BFFR	121
Table 6.1	Sample codes of AgNW-PAN hollow fibers used for supercapacitor fabrication	136
Table 6.2	Properties of selected AgNW-PAN hollow fibers	137

LIST OF SCHEMES

Scheme No.	Scheme caption	Page No.
Scheme 1.1	Representation of junction points in different nanostructures	4
Scheme 2.1	Schematic representation of the mechanism of synthesis of AgNWs via interfacial chemistry	40
Scheme 2.2	Schematic representation of AgNWs formation in glycerol mediated polyol synthesis	56
Scheme 3.1	Electrospinning set-up for making nanofiber web	73
Scheme 3.2	Schematic representation of alignment of AgNWs inside electrospun nanofibers	84
Scheme 4.1	Schematic of set-up used for making hollow fiber	90
Section 4.2	Schematic showing mechanism of conductive hollow fiber formation	97
Scheme 5.1	Scheme 5.1. Schematic showing response of conductive layer to deformation.	130
Scheme 6.1	Scheme showing fabrication of parallel assembled AgNW-PAN hollow conductive fibers along with a photograph of fabricated device	138
Scheme 6.2	Scheme showing formation of co-axial assembled AgNW-PAN hollow conductive fibers one having annulus filled with electrolyte and other having both core and annulus filled with electrolyte along with a photograph of fabricated device	139

Scheme 6.3	Schematic representing the distance between the electrodes in parallel and co-axial assembly	147
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List of Symbols and Abbreviations

Symbol/Abbreviation	Expanded Form/Term
EDOT	3,4-Ethylenedioxythiophene
Ag-Py	Ag-Pyridine
AFM	Atomic force microscope
BFFR	Bore fluid flow rate
BMI	Brain machine interface
CNFs	Carbon nanofibers
CNTs	Carbon nanotubes
CMC	Carboxymethyl cellulose
CTAB	Cetyltrimethyl ammonium bromide
CVD	Chemical vapor deposition
CuCl ₂	Copper chloride
DMF	Dimethylformamide
EDX	Energy dispersive X-ray
EG	Ethylene glycol
FeCl ₃	Ferric chloride
Fe(NO ₃) ₃	Ferric nitrate
FE-SEM	Field emission- scanning electron microscope
GNP	Graphite nanoplatelets
HDPE	High density polyethylene
MEV	Minimum electrospinning voltage
MTPs	Multiply twinned particles

MWNTs	Multi-walled nanotubes
NP	Nanoparticles
NWs	Nanowires
OLEDs	Organic light-emitting diodes
H ₃ PO ₄	Phosphoric acid
PEDOT: PSS	Poly(3,4-ethylenedioxythiophene):polystyrene sulphonate
PAN	Poly(acrylonitrile)
PET	Poly(ethylene terphthalate)
PVA	Poly(vinyl alcohol)
PVDF-HFP	Poly(vinylidene fluoride- <i>co</i> -hexafluoropropylene)
PVP	Poly(vinylpyrrolidone)
Pani	Polyaniline
PC	Polycarbonate
PDMS	Polydimethylsiloxane
PP	Polypropylene
Ppy	Polypyrrole
PS	Polystyrene
PU	Polyurethane
AgCl	Silver chloride
AgNP	Silver nanoparticle
AgNWs	Silver nanowires
AgNO ₃	Silver nitrate
SWCNTs	Single-walled carbon nanotubes

NaCl	Sodium chloride
NaOH	Sodium hydroxide
SBS	Styrene butadiene styrene
SEBS	Styrene-ethylene-butadiene-styrene
SERS	Surface enhanced Raman scattering
TEM	Transmission electron microscope
Fe(acac) ₃	Tris(aceylactonato) iron(III)
UV	Ultraviolet
ZnO	Zinc oxide
σ	Conductivity
G	Conductance