STUDIES ON SHAPE MEMORY-COUPLED-PIEZOELECTRIC NANOCOMPOSITES

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by

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Supreme Almighty

My grandfather (Late Santosh Kumar Sengupta)

Late Revered Swami Sagarananda ji Maharaj

My loving parents Smt. Silpi Chowdhury and

Late Goutam Chowdhury

Certificate

This is to certify that the thesis entitled "**Studies on shape memory-coupled-piezoelectric nanocomposites**" being submitted by **Mr**. **Anupam Chowdhury,** to the Indian Institute of Technology Delhi, for the award of the degree of **Doctor of Philosophy** in the Department of Textile and Fibre Engineering, is a record of bonafide research work carried out by him. Mr. Chowdhury has worked under our guidance and supervision and fulfilled all the requirements for the submission of the thesis.

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

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Anupam Chowdhury

Abstract

The recent times have witnessed an upsurge in growth of smart materials that can perform multiple functions within a single system. These materials can sense and respond to their environment, thereby making them indispensable for next generation technologies. There is a rising demand to probe further into the design of these materials particularly meant for sensing and actuation. Among many types of smart materials like electroactive polymers, magnetostrictive materials, self-healing systems, there are two interesting categories, i.e., combination of shape memory and piezoelectric materials which may be very useful for sensing and actuation. Their ability to respond to external stimuli surpasses them for exciting future applications.

The thesis deals with development of shape memory-coupled-piezoelectric nanocomposites, which was accomplished by (i) combining shape memory and piezoelectric polymer and (ii) combining shape memory polymer and piezoelectric filler. Therefore, by these two approaches, we can clearly distinguish the synergism between polymer-polymer and polymer-filler system. Initially, blends of polyurethane and PVDF was processed by melt extrusion to prepare filaments possessing higher relative piezoelectric β phase of 66 %. The alignment of polymer chains along the fibre axis during melt extrusion resulted in the transformation of α to β transformation of PVDF without additional stretching and poling. The open-circuit voltage and short-circuit current was 280 mV and 0.154 μA, respectively for the blends. The addition of optimum r-GO promoted α phase and increased conductivity leading to an open circuit voltage of 349 mV and short-circuit current of 0.257 μA. The strain recovery value of 100% and fixity of 43% was observed at 50/50 PU-PVDF blends, whereas recovery of 100% and fixity of 46% was observed for PU/PVDF-0.3% rGO nanocomposite blends. The crystallinity and morphology of piezoelectric fillers plays an important role in tuning electromechanical and piezoelectric properties of the nanocomposites. Therefore, $ZnSnO_3$(ZT) was chosen as the self-poled piezoelectric filler whose crystallinity was tuned primarily through varying concentration of NaOH and morphology was controlled through concentration of capping agent along with physical and chemical parameters. Lower mineralizer concentration promoted different polycrystalline species like SnO_2 and Zn_2SnO_4 exhibiting nanoparticle morphologies. Higher mineralizer concentration promoted the formation of face centred cubic zinc hydroxystannate phase, exhibiting ideal ferroelectric behaviour and promising piezoelectric behaviour. Further, tuning ((Zn^{2+} or Sn^{4+}) / urea) (m) promoted formation of high aspect ratio nanorods having aspect ratio of ~7.95 ± 2.55 at a fixed mineralizer concentration, fill volume, temperature and

time of the reaction. The thorough and systematic study was conducted to study the effect of physical and chemical parameters to obtain one step ZT nanorods. The nanorods synthesized at different m ratios were tested for ferroelectric, dielectric and piezoelectric properties. The high aspect nanorods had remnant polarization of 0.11 μC/cm^2, lossy ferroelectric behaviour and showed effective piezoelectric coefficient of 0.20 pm/V.

In the next part of the research, the ZT nanocubes synthesized using 0.25 M mineralizer and high aspect nanorods synthesized using m= 0.04 were used as the piezoelectric filler in SMPU matrix for the preparation of nanocomposites in the form of solvent cast films, melt spun fibres and electrospun nanofibres. The solvent cast nanocomposite films were prepared by using ZT nanocubes. The highest dielectric constant of 5.039 was shown by SMPU-5%ZT among all samples. PZ-5 displayed strain fixity of 88.89 % and strain recovery of 87.5 % at the end of 1st cycle due to the presence of filler, which further enhanced to 100% at the end of 2nd and 3rd cycle. PZ-5 shown highest peak-to-peak open-circuit voltage of 3.26 V, short-circuit current of 1.13 μA, charge measured in shielded and unshielded mode was 0.26 nC and 2.2 nC, and power density of ~ 2.12 mW/m^2 at 8.2 MΩ. The output of nanogenerator was used for charging a capacitor and lighting up an LED. The shape customized PZ-5 displayed potential applications as touch sensor and temperature sensors. The melt spun SMPU/ZT nanocomposite filaments were fabricated by employing ZT nanorods. It displayed open-circuit voltage and short-circuit current of ~3.69 V and ~0.52 μA), power density of 1.16 mW/m^2 at load resistance of 10 MΩ. The generated voltage was used to power LED, thereby indicating its practical real-life implementations. The shape memory behaviour of PZ-0.75 showed 100 % strain fixity and strain recovery at the end of 3rd cycle of repeated training of loading and unloading cycles. The multifunctional woven fabric made of PZ-0.75 was used to monitor wrist movements. The structure-property relationship of ZT nanocubes and nanorods was studied in electrospinning process. The interesting thermal transitions were observed for addition of fillers in SMPU matrix. The highest piezoelectric output was observed at 1% concentration of nanorods (peak-to-peak open-circuit voltage of ~ 2.20 V, short-circuit current of ~ 1.31 μA, power density of 2.6 mW/m^2 at 1 MΩ load resistance) and nanocubes (peak-to-peak open-circuit voltage of ~1.4 V, short-circuit current of ~1.25 μA, power density of ~1.2 mW/m^2 at 680 kΩ) in SMPU nanofibres. It showed promising real-life applications as multifunctional material responding to different flex movements of a finger and tracking repeated respiratory movements. It showed promising shape memory behaviour. Therefore, the developed multifunctional nanocomposites are promising in the field of sensors, actuators and energy harvesters.

सार

हाल के समय में ऐसे स्मार्ट पदार्थों के विकास में तेज़ी देखी गई है जो एक ही प्रणाली में कई कार्य कर सकते हैं। ये पदार्थ अपने परिवेश को महसूस कर सकते हैं और उस पर प्रतिक्रिया दे सकते हैं, जिससे ये अगली पीढ़ी की तकनीकों के लिए अपरिहार्य हो जाते हैं। इन पदार्थों के डिज़ाइन, विशेष रूप से संवेदन और क्रियान्वयन के लिए, और अधिक गहन अध्ययन की माँग बढ़ रही है। इलेक्ट्रोएक्टिव पॉलिमर, मैग्नेटोस्ट्रिक्टिव पदार्थ, स्व-उपचार प्रणालियाँ जैसे कई प्रकार के स्मार्ट पदार्थों में, दो दिलचस्प श्रेणियाँ हैं, अर्थात् आकार स्मृति और पीज़ोइलेक्ट्रिक पदार्थों का संयोजन, जो संवेदन और क्रियान्वयन के लिए बहुत उपयोगी हो सकते हैं। बाहरी उत्तेजनाओं पर प्रतिक्रिया करने की उनकी क्षमता भविष्य के रोमांचक अनुप्रयोगों के लिए उनसे कहीं बेहतर है।

यह शोध प्रबंध आकार स्मृति-युग्मित-पीजोइलेक्ट्रिक नैनोकंपोजिट के विकास से संबंधित है, जिसे (i) आकार स्मृति और पीजोइलेक्ट्रिक पॉलीमर के संयोजन और (ii) आकार स्मृति पॉलीमर और पीजोइलेक्ट्रिक फिलर के संयोजन द्वारा पूरा किया गया था। इसलिए, इन दो तरीकों से, हम पॉलीमर-पॉलिमर और पॉलीमर-फिलर प्रणाली के बीच तालमेल को स्पष्ट रूप से पहचान सकते हैं। प्रारंभ में, पॉलीयुरेथेन और PVDF के मिश्रणों को 66% के उच्च सापेक्ष पीजोइलेक्ट्रिक β चरण वाले फिलामेंट तैयार करने के लिए मेल्ट एक्सट्रूज़न द्वारा संसाधित किया गया था। मेल्ट एक्सट्रूज़न के दौरान फाइबर अक्ष के साथ पॉलीमर श्रृंखलाओं के संरेखण के परिणामस्वरूप अतिरिक्त स्ट्रेचिंग और पोलिंग के बिना PVDF का α से β रूपांतरण हुआ। इष्टतम r-GO के संयोजन से α चरण में वृद्धि हुई और चालकता में वृद्धि हुई, जिससे 349 mV का खुला परिपथ वोल्टेज और 0.257 μA का लघु-परिपथ धारा प्राप्त हुई। 50/50 PU-PVDF मिश्रणों में 100% विकृति पुनर्प्राप्ति मान और 43% स्थिरता देखी गई, जबकि PU/PVDF-0.3% rGO नैनोकंपोजिट मिश्रणों में 100% पुनर्प्राप्ति और 46% स्थिरता देखी गई। नैनोकंपोजिट के विद्युत-यांत्रिक और

पीजोइलेक्ट्रिक गुणों को समायोजित करने में पीजोइलेक्ट्रिक भरावों की क्रिस्टलीयता और आकारिकी महत्वपूर्ण भूमिका निभाती है। इसलिए, $ZnSnO_3$(ZT) को स्व-ध्रुवित पीजोइलेक्ट्रिक भराव के रूप में चुना गया, जिसकी क्रिस्टलीयता को मुख्य रूप से NaOH की बदलती सांद्रता के माध्यम से समायोजित किया गया और आकारिकी को भौतिक और रासायनिक मापदंडों के साथ कैपिंग एजेंट की सांद्रता के माध्यम से नियंत्रित किया गया। कम खनिज सांद्रता ने नैनोकणों की आकारिकी प्रदर्शित करने वाले SnO_2 और Zn_2SnO_4 जैसी विभिन्न पॉलीक्रिस्टलाइन प्रजातियों को बढ़ावा दिया। उच्च खनिज सांद्रता ने फलक केंद्रित क्यूबिक जिंक हाइड्रॉक्सीस्टैनेट चरण के निर्माण को बढ़ावा दिया, जिसने आदर्श फेरोइलेक्ट्रिक व्यवहार और आशाजनक पीजोइलेक्ट्रिक व्यवहार प्रदर्शित किया। एक-चरण ZT नैनोरॉड प्राप्त करने के लिए भौतिक और रासायनिक मापदंडों के प्रभाव का अध्ययन करने हेतु एक गहन और व्यवस्थित अध्ययन किया गया। विभिन्न Zn^{2+}/urea अनुपातों पर संश्लेषित नैनोरॉड्स का फेरोइलेक्ट्रिक, डाइइलेक्ट्रिक और पीजोइलेक्ट्रिक गुणों के लिए परीक्षण किया गया। उच्च अभिमुखता वाले नैनोरॉड्स में 0.11 $\mu C/cm^2$ का अवशिष्ट ध्रुवीकरण, हानिपूर्ण फेरोइलेक्ट्रिक व्यवहार और 0.20 pm/V का प्रभावी पीजोइलेक्ट्रिक गुणांक प्रदर्शित हुआ। शोध के अगले भाग में, 0.25 M मिनरलाइज़र का उपयोग करके संश्लेषित ZT नैनोक्यूब्स और m= 0.04 का उपयोग करके संश्लेषित उच्च आस्पेक्ट नैनोरॉड्स का उपयोग SMPU मैट्रिक्स में पीज़ोइलेक्ट्रिक फिलर के रूप में किया गया ताकि सॉल्वेंट कास्ट फ़िल्म्स, मेल्ट स्पन फ़ाइबर और इलेक्ट्रोस्पन नैनोफ़ाइबर के रूप में नैनोकंपोजिट तैयार किए जा सकें। सॉल्वेंट कास्ट नैनोकंपोजिट फ़िल्में ZT नैनोक्यूब्स का उपयोग करके तैयार की गईं। सभी नमूनों में SMPU-5%ZT ने 5.039 का उच्चतम परावैद्युत स्थिरांक दर्शाया। PZ-5 ने फिलर की उपस्थिति के कारण पहले चक्र के अंत में 88.89% की विकृति स्थिरता और 87.5% की विकृति पुनर्प्राप्ति प्रदर्शित की, जो दूसरे और तीसरे चक्र के अंत में 100% तक बढ़ गई। PZ-5 ने 3.26 V का उच्चतम पीक-टू-पीक ओपन-सर्किट वोल्टेज, 1.13 μA का शॉर्ट-सर्किट करंट, परिरक्षित और अप्रतिरक्षित मोड में मापा गया चार्ज 0.26 nC और 2.2 nC, और 8.2 MΩ

पर ~ 2.12 mW/m^2 का पावर घनत्व दिखाया। नैनोजनरेटर के आउटपुट का उपयोग एक संधारित्र को चार्ज करने और एक LED को प्रकाशित करने के लिए किया गया था। आकार अनुकूलित PZ-5 ने टच सेंसर और तापमान सेंसर के रूप में संभावित अनुप्रयोगों को प्रदर्शित किया। पिघले हुए SMPU/ZT नैनोकंपोजिट फिलामेंट्स को ZT नैनोरॉड्स का उपयोग करके तैयार किया गया था। इसने 10 MΩ के लोड प्रतिरोध पर 1.16 mW/m^2 का ओपन-सर्किट वोल्टेज और शॉर्ट-सर्किट करंट ~3.69 V और ~0.52 μA), पावर घनत्व 1.16 mW/m^2 प्रदर्शित किया। उत्पन्न वोल्टेज का उपयोग एलईडी को पावर देने के लिए किया गया था, जिससे इसके व्यावहारिक वास्तविक जीवन के कार्यान्वयन का संकेत मिलता है। PZ-0.75 के आकार स्मृति व्यवहार ने लोडिंग और अनलोडिंग चक्रों के दोहराया प्रशिक्षण के तीसरे चक्र के अंत में 100% तनाव स्थिरता और तनाव वसूली दिखाई। कलाई की गतिविधियों पर नज़र रखने के लिए PZ-0.75 से बने बहुक्रियाशील बुने हुए कपड़े का उपयोग किया गया था एसएमपीयू नैनोफाइबर में नैनोरॉड्स (~ 2.20 V का पीक-टू-पीक ओपन-सर्किट वोल्टेज, ~ 1.31 μA का शॉर्ट-सर्किट करंट, 1 MΩ लोड प्रतिरोध पर 2.6 mW/m^2 का पावर घनत्व) और नैनोक्यूब्स (~1.4 V का पीक-टू-पीक ओपन-सर्किट वोल्टेज, ~1.25 μA का शॉर्ट-सर्किट करंट, 680 kΩ पर ~1.2 mW/m^2 का पावर घनत्व) की 1% सांद्रता पर उच्चतम पीजोइलेक्ट्रिक आउटपुट देखा गया। इसने एक उंगली के विभिन्न फ्लेक्स मूवमेंट पर प्रतिक्रिया करने और बार-बार होने वाली श्वसन गतिविधियों को ट्रैक करने वाले बहुक्रियाशील पदार्थ के रूप में आशाजनक वास्तविक जीवन के अनुप्रयोगों को दिखाया। इसने आशाजनक आकार स्मृति व्यवहार दिखाया। इसलिए, विकसित बहुक्रियाशील नैनोकंपोजिट सेंसर, एक्चुएटर्स और ऊर्जा संग्राहकों के क्षेत्र में आशाजनक हैं।

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LIST OF ABBREVIATIONS

PZT	Lead Zirconate Titanate
$BaTiO_3$	Barium Titanate
KNN	Potassium Sodium Niobate
$ZnSnO_3$,ZT,ZS	Zinc Stannate
ZnO	Zinc Oxide
$BiFeO_3$	Bismuth Ferrite
SMA	Shape Memory Alloy
SMP	Shape Memory Polymer
PVDF	Polyvinylidene Fluoride
SMPU	Shape Memory Polyurethane
r-GO	Reduced Graphene Oxide
PU	Polyurethane
ATR-FTIR	Attenuated Total Reflection-Fourier Transform Infrared Spectroscopy
XRD	X-Ray Diffraction
UV-vis	Ultraviolet Visible
LED	Light Emitting Diode
CTS	Carpel Tunnel Syndrome
LN	Lithium Niobate
PVDF-TrFE	Polyvinylidene Difluoride-Trifluoroethylene
ZTO	Zinc Tin Oxide
FESEM	Field Emission Scanning Electron Microscope
PFM	Piezoresponse Force Microscopy
TGA	Thermogravimetric Analysis
TEM	Transmission Electron Microscopy
PE	Polarization-Electric Field
LCR	Inductance, Capacitance and Resistance
DSC	Differential Scanning Calorimetry
P_r	Remnant Polarization
$Bi_{0.5}Na_{0.5}TiO_3$	Sodium Bismuth Titanate
PT	Lead Titanate

$BiMeO_3$	Metal Oxides Of Bismuth
GaN	Galium Nitride
NaOH	Sodium Hydroxide
MoS_2	Molybdenum Sulphide
$KNbO_3$	Potassium Niobate
$AgNbO_3$	Silver Niobate
$NaNbO_3$	Sodium Niobate
$LiTaO_3$	Lithium Tantalate
eV	Electron Volts
MgO	Magnesium Oxide
BTO	Barium Titanate
PDMS	Polydimethyl Siloxane
MWCNT	Multiwalled Carbon Nanotube
TiO_2	Titanium Dioxide
PVC	Poly (Vinyl Chloride)
Nitinol	Nickel Titanium Alloy
T_g	Glass Transition Temperature
T_m	Melting Temperature
MWNT	Multiwalled Nanotubes
CNF	Carbon Nanofibre
SiC	Silicon Carbide
Ppy	Polypyrrole
POSS	Polyhedral Oligomeric Silsesquioxane
Ti-Ni-Cu	Titanium-Nickel-Copper
NdFeB	Neodymium-Iron-Boron
PLA	Polylactic Acid
PEA	Polyesteramide
AESO	Acrylate Epoxidized Soybean Oil
TMSPM	3-(Trimethoxysilyl) Propyl Methacrylate
PDA	Polydopamine
IR	Infrared
THDA	Trimethylhexamethylenediamine
PCL	Polycaprolactone

MDI	Methylene Diphenyl Diisocyanate
DUT	Device Under Test
MWS	Maxwell Wagner Sillars
AFM	Atomic Force Microscopy
DART	Dual AC Resonance Tracking Mode
DC	Direct Current
AC	Alternating Current
EDX	Energy Dispersive X-Ray
KBr	Potassium Bromide