SHEAR THICKENING FLUID ENCAPSULATION IN ELECTROSPUN UHMWPE AND ITS BALLISTIC PERFORMANCE UNDER HIGH STRAIN RATE

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by

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"Mind is like water,

When it is agitated, it becomes difficult to see,

But if you allow it to settle, the answer becomes clear."

– ***Master Oogway***

Dedicated To My

'Family'

CERTIFICATE

This is to certify that the thesis entitled, ***"Shear thickening fluid encapsulation in electrospun UHMWPE and its ballistic performance under high strain rate"*** *submitted by* ***Ms. Prajesh Nayak*** *to the* ***Indian Institute of Technology Delhi,*** *for the fulfilment of award of the degree,* ***Doctor of Philosophy,*** *is a record of bonafide research work carried out by her under our supervision and guidance. This thesis has been prepared in conformity with the rules and regulations of the Indian Institute of Technology Delhi, New Delhi.*

The thesis, in our opinion, is worthy of consideration for award of the degree of ***Doctor of Philosophy*** *in accordance with the regulations of the Institute. To the best of my knowledge, the results embodied in the thesis have not been submitted to any other University or Institute for the award of any other Degree or Diploma.*

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ABSTRACT

In this study, shear thickening fluid (STF) encapsulated electrospun ultra-high molecular weight polyethylene (UHMWPE) mat was prepared and its ballistic performance was evaluated at high strain rates under dynamic compressive loading. Two different molecular weights of UHMWPE, M_w=2.21×10^6 g/mole (UM2.21) and 4.43×10^6 g/mole (UM4.43), were used to prepare the solutions at different concentrations in a binary mixture of decalin and cyclohexanone (70:30 ratio) solvent. Using de-Gennes's scaling theory, a correlation was established between rheological properties and electrospun morphology of the solutions to identify their critical entanglement concentrations, the concentrations at which polymer molecules started to entangle with each other. A smooth fibrous morphology was obtained when the concentration of the solution reached to 2-2.5 times the entanglement concentration. Further work was carried out by choosing higher molecular weight UHMWPE (UM4.43) at its optimum smooth fiber solution concentration (2 wt.%) due to its better properties. Other electrospinning process parameters such as applied voltage, solution conductivity, solvent type, etc were also optimized for UM4.43.

In the next step, the stretching of electrospun mat of UM4.43 was performed inside a hot air oven at different temperature range from 60-150℃. These stretched fibers, along with a single fiber of Spectra® lamina (as a control sample), were characterized for their mechanical performance and morphological analysis by SEM, XRD and tensile test under quasi-static condition.

Shear thickening fluid was synthesized with nano silica particles and characterized for its rheological behavior such as steady strain rate, dynamic strain rate and structural regeneration thixotropy test. The ballistic performance of nonwoven fabric with STF was first evaluated using a commercially available nonwoven felt of UHMWPE. This hypothesis of non-woven felt behavior was utilized in electrospun nonwoven fabric of UHMWPE.

Before STF impregnation in the electrospun mat, the surface properties such as the low coefficient of friction of UHMWPE fibers were altered by solution blending of high-density polyethylene (HDPE) with UHMWPE to increase the adhesion of silica particles on the fiber surface. Electrospinning of UHMWPE (UM4.43) and HDPE blended solution was performed at three different weight ratio compositions (100:0, 67:33 and 50:50) for obtaining miscible blend compositions. Mat prepared at miscible blends composition of UHMWPE and HDPE (67:33) was selected for shear thickening fluid impregnation study. Lastly, STF impregnated electrospun UHMWPE/HDPE fabric and UHMWPE felt (as a control sample) were encapsulated firmly by enclosing them using stretched UHMWPE fiber and tested for its high strain rate behavior. The high strain rate performance of STF encapsulated samples showed a significant improvement in peak stress in both samples enclosed by stretched UHMWPE fiber as compared to the unenclosed neat felt sample. From this study it was found that STF encapsulation after its impregnation in the fabric can improve the ballistic performance by helping STF to perform synergistically with lab synthesised electrospun fabric and can be a precursor for further technology development.

सार

इस अध्ययन में, शीयर थिकनिंग फ्लुइड (एसटीएफ) एनकैप्सुलेटेड इलेक्ट्रोसपुन अल्ट्रा-हाई मॉलिक्यूलर वेट पॉलीइथाइलीन (यूएचएमडब्ल्यूपीई) मैट तैयार किया गया था और डायनेमिक कंप्रेसिव लोडिंग के तहत उच्च तनाव दरों पर इसके बैलिस्टिक प्रदर्शन का मूल्यांकन किया गया था। यूएचएमडब्ल्यूपीई के दो अलग-अलग आणविक भार, $Mw=2.21\times10^6$ (यूएम2.21) और 4.43×10^6 ग्राम/मोल (यूएम4.43), का उपयोग डेकालिन और साइक्लोहेक्सानोन के द्विआधारी मिश्रण में विभिन्न सांद्रता पर समाधान तैयार करने के लिए किया गया था। (70:30 अनुपात) विलायक। डी-जीन्स के स्केलिंग सिद्धांत का उपयोग करते हुए, उनके महत्वपूर्ण उलझाव सांद्रता की पहचान करने के लिए समाधानों के रियोलॉजिकल गुणों और इलेक्ट्रोस्पन आकारिकी के बीच एक सहसंबंध स्थापित किया गया था, जिस सांद्रता पर बहुलक अणु एक दूसरे के साथ उलझने लगे थे। एक चिकनी रेशेदार आकृति विज्ञान प्राप्त किया गया था जब समाधान की एकाग्रता उलझाव की एकाग्रता के 2-2.5 गुना तक पहुंच गई थी। इसके बेहतर गुणों के कारण इष्टतम चिकनी फाइबर समाधान एकाग्रता (2 wt%) पर उच्च आणविक भार यूएचएमडब्ल्यूपीई (यूएम4.43) को चुनकर आगे का काम किया गया। अन्य इलेक्ट्रोस्पिनिंग प्रक्रिया पैरामीटर जैसे लागू वोल्टेज, समाधान चालकता, विलायक प्रकार, आदि को भी यूएम4.43 के लिए अनुकूलित किया गया था।

अगले चरण में, यूएम4.43 के इलेक्ट्रोस्पन मैट को 60-150 °C से अलग तापमान रेंज में गर्म हवा के ओवन के अंदर खींचा गया था। स्पेक्ट्रा® लेमिना (एक नियंत्रण नमूने के रूप में) के एक एकल फाइबर के साथ इन फैले हुए तंतुओं को अर्ध-स्थैतिक स्थिति के तहत एसईएम, एक्सआरडी और तन्यता परीक्षण द्वारा उनके यांत्रिक प्रदर्शन और रूपात्मक विश्लेषण के लिए चित्रित किया गया था।

कतरनी गाढ़ा द्रव नैनो सिलिका कणों के साथ संश्लेषित किया गया था और इसके रियोलॉजिकल व्यवहार जैसे स्थिर तनाव दर, गतिशील तनाव दर और संरचनात्मक पुनर्जनन थिक्सोट्रॉपी परीक्षण के लिए विशेषता थी। एसटीएफ के साथ गैर बुने हुए कपड़े के बैलिस्टिक प्रदर्शन का मूल्यांकन पहले यूएचएमडब्ल्यूपीई

के व्यावसायिक रूप से उपलब्ध गैर बुने हुए महसूस का उपयोग करके किया गया था। गैर बुने हुए महसूस किए गए व्यवहार की इस परिकल्पना का उपयोग यूएचएमडब्ल्यूपीई के इलेक्ट्रोस्पन गैर बुने हुए कपड़े में किया गया था।

इलेक्ट्रोसपन मैट में एसटीएफ संसेचन से पहले, यूएचएमडब्ल्यूपीई फाइबर के घर्षण के कम गुणांक जैसे सतह गुणों को फाइबर सतह पर सिलिका कणों के आसंजन को बढ़ाने के लिए यूएचएमडब्ल्यूपीई के साथ उच्च घनत्व पॉलीथीन (एचडीपीई) के मिश्रण से बदल दिया गया था। यूएचएमडबल्यूपीई (यूएम4.43) और एचडीपीई ब्लेंडेड सॉल्यूशन की इलेक्ट्रोस्पिनिंग तीन अलग-अलग वेट रेशियो कंपोज़िशन (100:0, 67:33 और 50:50) पर की गई थी ताकि मिसिबल ब्लेंड कंपोज़िशन प्राप्त किया जा सके। यूएचएमडबल्यूपीई और एचडीपीई (67:33) के गलत मिश्रण मिश्रण पर तैयार चटाई को कतरनी गाढ़ा द्रव संसेचन अध्ययन के लिए चुना गया था। अंत में, एसटीएफ ने इलेक्ट्रोसपुन यूएचएमडब्ल्यूपीई/ एचडीपीई फैब्रिक और यूएचएमडब्ल्यूपीई महसूस किया (एक नियंत्रण नमूने के रूप में) उन्हें फैलाए गए यूएचएमडब्ल्यूपीई फाइबर का उपयोग करके मजबूती से घेर लिया गया और इसके उच्च तनाव दर व्यवहार के लिए परीक्षण किया गया। एसटीएफ एनकैप्सुलेटेड नमूनों के उच्च तनाव दर प्रदर्शन ने यूएचएमडब्ल्यूपीई फाइबर से घिरे दोनों नमूनों में बिना साफ-सुथरे महसूस किए गए नमूने की तुलना में चरम तनाव में महत्वपूर्ण सुधार दिखाया। इस अध्ययन से यह पाया गया कि कपड़े में संसेचन के बाद एसटीएफ एनकैप्सुलेशन, एसटीएफ को प्रयोगशाला संश्लेषित इलेक्ट्रोस्पन कपड़े के साथ सहक्रियात्मक रूप से प्रदर्शन करने में मदद करके बैलिस्टिक प्रदर्शन में सुधार कर सकता है और आगे के प्रौद्योगिकी विकास के लिए अग्रदूत साबित हो सकता है।

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List of Abbreviations

Abbreviation	Description
AFM	Atomic force microscopic
CA	Cellulose Acetate
CNT	Carbon nanotube
DLS	Dynamic light scattering
DMAC	N, N-dimethylacetamide
DMF	N, N-dimethylformamide
DSC	Differential scanning calorimetry
EG	Ethylene glycol
FTIR	Fourier transform infrared spectroscopy
HDPE	High-Density Polyethylene
HMPP	High modulus polypropylene
iPP	Isotactic polypropylene
IR	Infrared
LLDPE	Linear low-density polyethylene
LVR	Linear viscoelastic region
NCD	Needle tip to collector distance
o-DCB	O-dichlorobenzene
PAN	Polyacrylonitrile
PCL	Polycaprolactone
PDLA	Poly(d-lactic acid)
PDLLA	D,L-poly(lactic acid)
PEG	Polyethylene glycol
PEO	Polyethylene oxide
PMMA	Poly(methyl methacrylate)
PP	Polypropylene
PPG	Polypropylene glycol
PSF	Polysulfone
PUU	Polyurethane urea
PVA	Polyvinyl alcohol
PVC	Poly (Vinyl Chloride)

PVDF	Polyvinylidene fluoride
PVP	Poly(vinylpyrrolidone)
RMS	Root mean square
SD	Standard Deviation
SEM	Scanning electron microscopy
SHB	Split-Hopkinson Bar
SHPB	Split-Hopkinson Pressure Bar
SQ	Sodium oleate
STA	Simultaneous thermal analysis
STF	Shear thinning fluid
STG	Shear thickening gel
TGA	Thermogravimetric analysis
THF	Tetrahydrofuran
UHMWPE	Ultra-high molecular weight polyethylene
UM2.21	UHMWPE of 2.21 million g/mole molecular weight
UM4.43	UHMWPE of 4.43 million g/mole molecular weight
UMHD0	Neat UHMWPE (100:0)
UMHD1	UHMWPE/HDPE (67:33)
UMHD2	UHMWPE/HDPE (50:50)
WAXD	Wide-angle X-ray diffraction analysis
XRD	X-ray diffraction analysis

List of Symbols

Symbol	Description
2θ	X-ray scattering angle
σ	Electric conductivity/Tensile strength
σs	Coefficient surface tension
σ(t)	Average stress in the sample
μ	Viscosity of suspending medium
λ	Wavelength of the X-ray source
ΔHm	Melt enthalpy
$\Delta H^0 m$	Melt enthalpy of the 100% crystalline polymer
A_S and A_B	Cross-section area of the sample and bars
β	Full-Width Half Maxima (radians)
c*	Overlap concentration
c_e	Entanglement concentration
c_0	Elastic wave velocity
D	Diffusion coefficient/Pore diameter/Fiber diameter/Crystallites size
D_b	Diameter of the bars
ε	Electric permittivity/elongation at break
ε_0	Permeability of vacuum
ε(t)	Strain rate within the sample
ε_i	Strain due to incident waves
ε_r	Strain due to reflected waves
ε_t	Strain due to transmitted waves
E	Elastic modulus
F_1	Force applied to the sample by the incident bar
F_2	Force applied to the sample by the transmitted bar
g	Gravitational acceleration
G' and G"	Storage and loss modulus
H	Distance between the needle tip to the collector
H_s	Sample thickness
k and K	Boltzmann's constant and Scherrer constant
L	Length of the needle tip

m	Consistency
M_w	Molecular weight of the polymer
n	Power-law index
[η]	Intrinsic solution viscosity
η_s	Solvent viscosity
η_0	Zero-shear viscosity
η_{sp}	Specific viscosity
Nav	Avogadro number
ρ	Mass density of liquid
ρ_p	Densities of polymers
ρ_s	Densities of 1-octanol
p	Differential pressure
Pe	Pèclet number
Q	Electrostatic charge
r	Radius of the suspended drop
R	Radius of droplet/Gas constant/Needle radius
$\langle R^2 \rangle^{1/2}$	Root mean squared end-to-end distance
R_a	Arithmetic mean roughness
R_g	Radius of gyration
R_h	Hydrodynamic radius
R_q	RMS roughness
T	Temperature
u_1 and u_2	Incident and transmission bar end displacement
ν	Flory's constant
W_w	Weight of the wet mat
W_d	Weight of the dry mat
χ	Flory-Huggins parameter
X_c	Degree of crystallinity
γ	Surface tension of fluid
$\dot{\gamma}$ and $\dot{\gamma}_c$	Shear rate and Critical shear rate
Z	Acoustic impedance of the material

Preamble

Background and motivation

Ballistic protective clothing is required to protect personnel from bullets and fragments. High-performance fibers, namely *p*-aramid-based fibers (Kevlar, Twaron) and ultra-high molecular weight polyethylene (UHMWPE) are specially engineered for ballistic armor, which works as a backbone of today's ballistic armor. However, enhanced sophisticated weapons and war strategy need more excellent threat protection, making present day armor cumbersome. Concerning this area, fabrics are treated with non-Newtonian shear thickening fluid to improve armor flexibility by reducing the number of layers for the same level of protection. Developing lightweight and strong materials for armor have always been an open area for research.

High-performance fibers possess unique properties due to their different base polymer and special fiber manufacturing processes which influence the microscopic structure and chain orientation of fibers. UHMWPE contain an extremely long chain of ethylene molecules which significantly contributes to its strength. In the main ethylene chain, the atoms are bonded with strong covalent bonds, while weak van der Waals bonds are present in the parallel orientation of the molecular chain. Because of its large molecular chain, a long overlap exists among the chain length. Consequently, the cumulative effect of van der Waals bonds is significant over the entire chain, which assists in bearing an enormous shear force between the molecules [1].

UHMWPE fibers produced by gel spinning techniques possess a high tensile strength of around 5 GPa. It is obtained by post-drawing of extruded filament at high temperatures and at different draw ratios [2]. However, it is far from the theoretical tensile strength of 32 GPa and modulus values of 324 GPa of perfect polyethylene crystals of C-C bond strength. Smook et al.[3] suggested that a perfect polyethylene crystal is possible to attain by preparing an extremely thin filament because the actual strength of the fiber is obtained by the lateral bonding between

the molecule [4]. Several methods have been used to make UHMWPE fiber with improved properties by orienting and aligning the molecules [5], [6].

The electrospinning process produces polymeric fibers with diameters varying from nano to several micro-meter ranges, that can possess unique properties like an immensely high surface area to volume ratio and outstanding mechanical properties as compared to larger diameter fibers of the same materials, which motivated this research to fabricate electrospun fibers of UHMWPE. However, the processing of UHMWPE is challenging due to the high melt viscosity and insolubility in common solvents at room temperature. It is only soluble in hydrocarbon solvents such as decalin, p-xylene, paraffin oil, etc., at their theta temperature. Therefore, a high-temperature solution electrospinning process was required to produce ultra-fine fibers of UHMWPE. Moreover, mechanical properties and fibers morphology was controlled by varying spinning processing parameters such as solution rheology, applied voltage, solution concentration, etc. These parameters were needed to be optimized for obtaining the processing window for defect-free uniform fiber of UHMWPE. However, the fully extended orientation of the molecular chain was challenging to achieve only a the single-step electrospinning method due to the high entanglement of UHMWPE chains. This behavior of electrospun fibers created second motivation for improving its performance further. Therefore, a post-processing of electrospun UHMWPE fiber was performed at a high temperature with load, which was further needed to be analyzed for its properties evolvement as compared to Spectra® fibers.

Rheological properties play a significant role in the spinning of fiber solution, which influences the productivity of fibers. High solution viscosity of UHMWPE leads to lower fiber productivity, but it improves by increasing the flowability of the solution by blending with low molecular weight polyethylene, i.e., HDPE. This was the third motivation to increase fiber productivity and determine the morphology of blended fibers, its effect of blending on the

productivity rate, etc. Encapsulation of STF inside an electrospun mat was the fourth motivation for this thesis work. To achieve this task, first, selecting the right nonwoven electrospun mat for STF impregnation, which can absorb a higher amount of STF was done based on fiber surface morphology and pore size distribution inside a mat which was enclosed inside the high temperature stretched electrospun fabric. For evaluation and comparison of properties of electrospun samples, nonwoven felt of UHMWPE was used. To understand ballistic performance of these samples, it was tested at a high strain rate (10^2-10^4) in dynamic loading conditions. High strain rate testing was performed using in-house fabricated Split-Hopkinson pressure bars for encapsulated and non-encapsulated electrospun mat and non-woven felt leading to better understanding of its application in future armor technology.