

**STUDIES ON HIGH DENSITY POLYETHYLENE / ULTRA
HIGH MOLECULAR WEIGHT POLYETHYLENE BLENDS
AND THEIR HYDROXYAPATITE FILLED COMPOSITES**

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**CENTRE FOR POLYMER SCIENCE AND ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY DELHI
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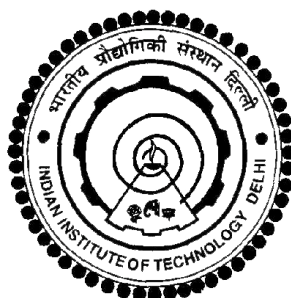
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Submitted

**In fulfillment of the requirements of the degree of
Doctor of Philosophy**

to



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CERTIFICATE

This is to certify that the thesis entitled “**Studies on High Density Polyethylene / Ultra High Molecular Weight Polyethylene Blends and Their Hydroxyapatite Filled Composites**” being submitted by **Mr. Harjeet Singh Jaggi**, to the Indian Institute of Technology Delhi, for the award of the degree of **Doctor of Philosophy** in the Centre for Polymer Science and Engineering, is a record of bonafide research work carried out by him. Mr. Jaggi has worked under my 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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Harjeet Singh Jaggi

Abstract

Binary blends of HDPE/UHMWPE were prepared by melt-mixing technique in a twin screw extruder in the composition range of 0-40 wt.% of UHMWPE. Test specimens were fabricated by injection/compression molding and examined for their thermal, rheological, morphological, quasi-static and dynamic mechanical responses. Furthermore, the use of essential work of fracture (EWF) approach adds a new dimension to the material development process. This EWF method critically assesses the resistances to crack initiation and crack propagation values responsible for energy dissipation in inner fracture process zone (IFPZ) and outer plastic deformation zone (OPDZ) respectively. The kinetic and energy related fracture parameters for example crack extension (Δa), crack tip opening displacement (CTOD), crack velocity (CTOD rate) and J-integral were evaluated to understand the rate sensitivity and crack resistance behavior of the blends. In addition, strain field analysis via digital image correlations techniques was performed to assess the strain distribution, which indirectly deciphers the stress energy dissipation ability and thus provides insight details about the fracture toughness. The microstructural changes due to UHMWPE incorporation in the HDPE matrix and its influence on the mechanical properties of investigated blends were comprehensively studied. It has been found that the blend containing 30 wt.% of UHMWPE possessed optimum tensile properties, impact strength and fracture resistance and hence, it was selected as the base matrix for the fabrication of HAP filled ternary composites.

Composites based on optimized blend matrix i.e., HDPE/UHMWPE (70/30 wt./wt.) and HAP filler were fabricated and assessed for their microstructural, viscoelastic and quasi-static mechanical responses. The morphological attributes such as state of dispersion of HAP and UHMWPE in the continuous HDPE matrix were characterized by using scanning electron

microscopy (SEM). The microstructural attributes, such as crystalline organization and fractured surface topography of the investigated blends and composites, were characterized by differential scanning calorimetry (DSC), wide-angle X-ray diffraction (WAXD) and other supporting techniques. The thermo-mechanical response of the blends and composites were studied by dynamic mechanical analysis (DMA). The melt rheology of blends and composites at low and high shear rates were studied by parallel plate rheometry and capillary rheometry respectively, to assess the frequency dependence of moduli and viscosities. Comparative assessment of the crack toughness behavior of the blends and composites were carried out following the EWF approach based on post yield fracture mechanics (PYFM) concept and J-integral based elastic plastic fracture mechanics (EPFM) approach. Crack propagation kinetics and strain field analysis of investigated blends and composites were eventually carried out for the better understanding of fracture mechanism.

Thus, our study establishes the material design ideology of stiffness-toughness enhancement via UHMWPE incorporation into HDPE matrix by manipulating the compositional ratio. Moreover, the use of energy based EWF and *J*-integral approaches add a new dimension to the material development process. Eventually, the study demonstrates the quantification of the stress wave dissipation modes via strain field analysis and strain mapping via multi-stage section diagrams, which is fundamentally a new approach not only to understand fracture mechanics for designing materials for specific applications but also to elucidate the validity regime of the post yield fracture mechanics principles for such model systems.

सार

एचडीपीई / यूएचएमडब्ल्यूपीई के द्विआधारी मिश्रणों को एक जुड़वां पेंच में पिघल-मिश्रण तकनीक द्वारा तैयार किया गया था यूएचएमडब्ल्यूपीई के 0-40% की संरचना रेंज में एक्सट्रूडर टेस्ट नमूने गढ़े थे इंजेक्शन / संपीड़न मोल्डिंग द्वारा और उनके थर्मल, रियोलॉजिकल, रूपात्मक, अर्ध-स्थिर और गतिशील यांत्रिक प्रतिक्रियाएं इसके अलावा, आवश्यक कार्य का उपयोग फ्रैक्चर (ईडब्ल्यूएफ) दृष्टिकोण सामग्री विकास प्रक्रिया में एक नया आयाम जोड़ता है। यह ईडब्ल्यूएफ विधि गंभीर रूप से दिक्कतों को दरकिनार करने के लिए प्रतिद्वंद्विता का मूल्यांकन करती है और प्रचार के मूल्यों को दरकिनार करता है आंतरिक फ्रैक्चर प्रोसेस ज़ोन (आईएफपीजेड) और बाहरी प्लास्टिक में ऊर्जा अपव्यय के लिए जिम्मेदार विरूपण क्षेत्र (ओपीडीजेड) क्रमशः। काइनेटिक और ऊर्जा संबंधित फ्रैक्चर पैरामीटर के लिए उदाहरण दरार एक्सटेंशन (Δa), दरार टिप खोलने विस्थापन (सीटीओडी), दरार वेग (सीटीओडी दर) और जे-इंटिग्रल का मूल्यांकन दर संवेदनशीलता और दरार प्रतिरोध को समझने के लिए किया गया था मिश्रणों का व्यवहार इसके अलावा, डिजिटल छवि सहसंबंध तकनीकों के माध्यम से तनाव क्षेत्र विश्लेषण तनाव वितरण का आकलन करने के लिए किया गया था, जो परोक्ष रूप से तनाव ऊर्जा को उजागर करता है अपव्यय क्षमता और इस तरह फ्रैक्चर बेरहमी के बारे में जानकारी प्रदान करता है। इस एचडीपीई मैट्रिक्स में यूएचएमडब्ल्यूपीई निगमन के कारण माइक्रोस्ट्रॉक्चरल परिवर्तन और इसके प्रभाव पर जांच किए गए मिश्रणों के यांत्रिक गुणों का व्यापक अध्ययन किया गया। यह किया गया है पाया कि 30% यू.एच.एमडब्ल्यू.पी.टी. युक्त मिश्रण में इष्टतम तन्यता वाले गुण हैं, प्रभाव शक्ति और फ्रैक्चर प्रतिरोध और इसलिए, इसे आधार मैट्रिक्स के रूप में चुना गया था एचएपी भरा हुआ टर्नरी कंपोजिट का निर्माण ऑप्टिमाइज्ड मिश्रण मैट्रिक्स पर आधारित कंपोजिट, अर्थात् एचडीपीई / यूएचएमडब्ल्यूपीई (70/30 वेट। / वाइट।) और एचएपी भराव उनके माइक्रोस्ट्रॉक्चर, विज़ोलास्टिक और अर्ध-स्थैतिक यांत्रिक प्रतिक्रियाओं के

लिए तैयार किए गए थे। निरंतर एचडीपीई मैट्रिक्स में एचएपी और यूएचएमडब्ल्यूपीई के फैलाव के राज्य जैसे रूपवाचक गुणों को स्कैनिंग इलेक्ट्रॉन माइक्रोस्कोपी (एसईएम) का उपयोग करके चित्रित किया गया था। क्रिस्टलीय संगठन और जांच किए गए मिश्रणों और कंपोजिट के खंडित सतह स्थलाकृति जैसे माइक्रोस्ट्रक्चरल विशेषताओं को अंतर स्कैनिंग कैलोरीमेट्री (डीएससी), चौड़े कोण एक्स-रे विवर्तन (डब्ल्यूएक्सडी) और अन्य सहायक तकनीकों की विशेषता थी। मिश्रणों और कंपोजिट के थर्मो-यांत्रिक प्रतिक्रिया का अध्ययन गतिशील मैकेनिकल विश्लेषण (डीएमए) द्वारा किया गया। मॉड्यूल और चिपचिपाहट की आवृत्ति निर्भरता का आकलन करने के लिए, कम और ऊंची कतरनी दरों पर मिश्रणों और कंपोजिट की पिंडली क्रमशः समानांतर प्लेट रीलमेट्री और केशिका रेमंडम द्वारा अध्ययन किया गया। मिश्रणों और कंपोजिट के दरार क्रूरता के व्यवहार के तुलनात्मक आकलन, ईजबूम के बाद के फ्रैक्चर यांत्रिकी (पीवायएफएम) अवधारणा और जे-इंटीग्रल आधारित इलास्टिक प्लास्टिक फ्रैक्चर यांत्रिकी (ईपीएफएम) दृष्टिकोण के आधार पर ईडब्ल्यूएफ दृष्टिकोण के बाद किया गया। फ्रैक्चर तंत्र की बेहतर समझ के लिए जांच कराए गए कैनेटीक्स और क्रैन फील्ड विश्लेषण में जांच की गई मिश्रणों और कंपोजिट का क्रैक प्रचार किया गया। इस प्रकार, हमारा अध्ययन रचनात्मक अनुपात को जोड़कर एचडीपीई मैट्रिक्स में यूएचएमडब्ल्यूपीई निगमन के माध्यम से कठोरता-कठोरता वृद्धि के भौतिक डिजाइन विचारधारा को स्थापित करता है। इसके अलावा, ऊर्जा आधारित ईडब्ल्यूएफ और जे-अभिन्न दृष्टिकोण का उपयोग सामग्री के विकास की प्रक्रिया में एक नया आयाम जोड़ते हैं। आखिरकार, अध्ययन मस्तिष्क-स्टेज खंड आरेखों के माध्यम से तनाव क्षेत्र विश्लेषण और तनाव के मानचित्रण के माध्यम से तनाव की लहर अपव्यय मोड की मात्रा का ठहराव दर्शाता है, जो कि न केवल विशिष्ट अनुप्रयोगों के लिए सामग्री को डिजाइन करने के लिए फ्रैक्चर यांत्रिकी को समझने के लिए बल्कि नए तरीके से स्पष्ट करने के लिए ऐसी मॉडल प्रणालियों के लिए उपज फ्रैक्चर यांत्रिकी सिद्धांतों के वैधता शासन।

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List of abbreviations and symbols

AFM	-	Atomic Force Microscopy
ASTM	-	American Society for Testing and Materials
CaCO ₃	-	Calcium carbonate
CTOD	-	Crack-tip opening displacement
DENT	-	Double-edge-notched-tension
DIC	-	Digital image correlation
DMA	-	Dynamic mechanical analysis
DPIM	-	Dynamic packing injection molding
DSC	-	Differential scanning calorimeter
$d\delta/dt$	-	CTOD rate
E	-	Young's modulus
E'	-	Storage modulus
E''	-	Loss modulus
EPDM	-	Ethylene propylene diene monomer rubber
EPDM-g-MA	-	Ethylene propylene diene grafted maleic anhydride
EPFM	-	Elastic-plastic fracture mechanics
EPR	-	Ethylene propylene rubber
EPR-g-MA	-	Ethylene propylene rubber grafted maleic anhydride
ESCR	-	Environmental stress cracking resistance
EVA	-	Ethylene vinyl acetate
EWf	-	Essential work of fracture
FPZ	-	Frontal process zones
FTIR	-	Fourier transform infra red spectroscopy
FWHM	-	Full width half maxima
GB	-	Glass bead
G'	-	Melt storage modulus
G''	-	Melt loss modulus
HAP	-	Hydroxyapatite
HDPE	-	High density polyethylene
IFPZ	-	Inner fracture process zone
i-PP	-	Isotactic polypropylene
J_c	-	Critical J -integral value
K	-	Stress-intensity factor

KCP	-	Kinetics of crack propagation
K_c	-	Critical stress-intensity factor
l	-	Ligament length
LDPE	-	Linear density polyethylene
LEFM	-	Linear elastic fracture mechanics
LLDPE	-	Linear low density polyethylene
LPE	-	Linear polyethylene
MA	-	Maleic anhydride
MFI	-	Melt flow index
NA	-	Nucleating agent
NBR	-	Butadiene-acrylonitrile rubber
N-EWF	-	Non-essential work of fracture
NRP	-	Natural rubber powder
OPDZ	-	Outer plastic deformation zone
PA	-	Polyamide
PC	-	Polycarbonate
PE	-	Polyethylene
POE	-	Polyethylene octene
POM	-	Polarized optical microscopy
PP	-	Polypropylene
PP-g-MA	-	Polypropylene grafted maleic anhydride
PYFM	-	Post-yield fracture mechanics
SEM	-	Scanning electron microscopy
SENT	-	Single-edge-notched-tension
SFA	-	Strain field analysis
s-PP	-	Syndiotactic polypropylene
TCP	-	Tri-calcium phosphate
$Tan \delta$	-	Tangent delta
T_c	-	Crystallization temperature
TEM	-	Transmission electron microscopy
T_g	-	Glass transition temperature
TGA	-	Thermogravimetric analysis
T_m	-	Melting temperature
TPE	-	Thermoplastics elastomer

TREF	-	Temperature rising elution fractionation
TTS	-	Time temperature superposition
UHMWPE	-	Ultra high molecular weight polyethylene
WAXD	-	2D Wide Angle X-ray Diffraction
w_e	-	Essential work of fracture
W_f	-	Total work of fracture
w_f	-	Specific total work of fracture
X_c	-	Degree of crystallinity
XRD	-	X-ray diffraction
β	-	Plastic zone shape factor
βw_p	-	Non-essential work of fracture
δ	-	Crack-tip opening displacement
Δa	-	Crack-extension
ΔH	-	Ideal enthalpy of melting a perfect crystal
ΔH_f	-	Measured enthalpy of melting
ε_b	-	Elongation at break
ρ	-	Density
σ_s	-	Tensile strength
σ_y	-	Yield stress