

**MORPHOLOGICAL, THERMO-MECHANICAL AND FRACTURE
PERFORMANCE OF POLYAMIDE-612/POLY(ETHYLENE-CO-
OCTENE) ELASTOMER BLENDS AND THEIR HALLOYSITE
NANOTUBE FILLED TERNARY NANOCOMPOSITES**

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

SUNIL KUMAR

Centre for Polymer Science and Engineering

Submitted

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to the



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CERTIFICATE

This is to certify that the thesis entitled “**Morphological, thermo-mechanical and fracture performance of polyamide-612/poly(ethylene-co-octene) elastomer blends and their halloysite nanotube filled ternary nanocomposites**” being submitted by **Mr. Sunil Kumar**, 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. Sunil Kumar 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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Sunil Kumar

ABSTRACT

Blends of polyamide-612 (PA-612)/poly(ethylene-co-octene)-grafted-maleic anhydride elastomer (POE-g-MA) were prepared in a twin screw extruder in the composition range of 0-35 wt% of POE-g-MA and were characterized for their morphological, thermo-kinetics, rheological, mechanical properties and fracture performance using SEM, DSC, TGA, tensile, flexural, Izod impact testing and essential work of fracture (EWF). The micro-structural changes due to POE-g-MA incorporation in the PA-612 matrix have been analyzed and their microstructural attributes-mechanical performance correlations have been explored for PA-612/POE-g-MA model system. Micromechanical aspects were analyzed using conventional theoretical models for low strain mechanical response such as rule of mixtures and foam model and high strain mechanical response such as Nicolais-Narkis model and porosity model.

The blend with 10 wt% of POE-g-MA showed optimum tensile, impact properties and fracture resistance and hence was selected as the (impact modified PA-612) base matrix for the preparation of halloysite nanotube (HNT) filled composites. Ternary nanocomposites of PA-612/POE-g-MA/HNT based on optimized blends of PA-612/POE-g-MA were prepared and characterized for their structural, rheological, thermal, mechanical and fracture behavior. The morphological attributes such as state of dispersion as a function of HNT content and *soft* elastomer phase domain sizes dispersed in PA-612 matrix were characterized by TEM and SEM, whereas microstructural attributes such as crystalline organization/chemical interaction and fractured surface topography of the optimized blends and their nanocomposites were characterized by WAXD, SEM and other supporting techniques. Non-isothermal crystallization kinetics behaviors of neat PA-612, PA-612/POE-g-MA blends and their nanocomposites were investigated by DSC vis-à-vis the determination of various crystallization parameters using

Avrami kinetics and Mo theory based models. The activation energies responsible for crystallization process have been estimated for the blends and their nanocomposites using Takhor equation. The thermo-mechanical response of the blends and nanocomposites were studied using dynamic mechanical analysis.

The melt rheology of blends and nanocomposites were studied by using parallel plate rheometry and capillary rheometry to assess the frequency dependence of moduli and complex viscosities.

Comparative assessment of the crack toughness behavior of the blends and nanocomposites were carried out following the essential work of fracture (EWF) approach based on post yield fracture mechanics (PYFM) concept. The kinetic and energy related fracture parameters like crack extension, crack tip opening displacements (CTOD) and crack velocity (CTOD rate) were used to evaluate the rate sensitivity of the blends and nanocomposites, while real-time visualization of strain field attributes responsible for toughness variations (as obtained by essential work of fracture approach) were computed based on surface displacement measurement techniques.

Firstly, our study establishes the material design ideology of toughness-enhancement via POE-g-MA incorporation into PA-612 matrix by manipulating the compositional ratio and hence the *soft*-phase domain size. Secondly, 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.

Contents

	Page No.
Certificate	
Acknowledgements	
Abstract	i-ii
Contents	iii-viii
List of Figures	ix-xiii
List of Tables	xiv-xv
Chapter-1: Introduction and literature survey	1-49
1.1. Overview of the chapter	1
1.2. Literature overview	1
1.2.1. Polyamides and toughened polyamides blends	1
1.2.2. Rheological response of polyamide blends	9
1.2.3. Crystallization behavior of blends	11
1.2.4. Fracture behavior of blends	13
1.2.5. Filled and fiber reinforced polyamide composites	16
1.2.6. Polymer/halloysitenanocomposites	17
1.2.7. Processing strategies for the fabrication of polymer/halloysitenanocomposites	19
1.2.8. Mechanical reinforcement of polyamides nanocomposites	22
1.2.9. Rheological response of nanocomposites	27
1.2.10. Crystallization behavior of nanocomposites	28
1.2.11. Fracture behavior of polyamide based micro/nano composites	29

1.3.	Motivation	32
1.4.	Objective of the present study	36
1.5.	Thesis format	37
	References	40-49
	Chapter-2: Experimental: materials and methods	50-63
2.1.	Overview of the chapter	50
2.2.	Materials and methods	50
2.2.1.	Raw materials	50
2.2.2.	Preparation of PA-612/POE-g-MA blends	51
2.2.3.	Fabrication of ternary nanocomposites	52
2.3.	Characterization and evaluation techniques	53
2.3.1.	Wide angle X-ray diffraction (WAXD)	53
2.3.2.	Morphological characterization	53
2.3.2.1	Cryo-fractured surface morphology	53
2.3.2.2	Ultra-microtomy and transmission electron microscopy (TEM)	53
2.3.3.	Rheological response	54
2.3.3.1	Determination of melt flow index (MFI)	54
2.3.3.2	Parallel plate rheology	54
2.3.3.3	Capillary rheology	54
2.3.4.	Thermal characterization	55
2.3.4.1	Differential scanning calorimetry (DSC)	55
2.3.4.2	Non-isothermal crystallization kinetics: DSC	55
2.3.4.3	Thermo-gravimetric analysis (TGA)	56

2.3.5. Mechanical properties	56
2.3.5.1 Dynamic mechanical analysis	56
2.3.5.2 Tensile properties	56
2.3.5.3 Flexural properties	56
2.3.5.4 Impact properties	57
2.3.6. Fracture mechanics	57
2.3.6.1 Essential work of fracture (EWF) measurements	57
2.3.6.2 Fracture kineticsparameters/crack resistance curves	59
2.3.6.3 Strain field evolution methodology	60
2.3.7. Fractured surface morphology by scanning electron microscopy	62
References	63
Chapter-3: Morphological, rheological and thermal properties of	64-91
PA-612/POE-g-MA blends	
3.1. Overview of the chapter	64
3.2. Structural characterization	64
3.3. Cryo-fractured surface morphology and phase distribution	65
3.4. Rheological behavior of blends	67
3.4.1. Melt flow index (MFI)	67
3.4.2. Low shear melt rheology (Parallel plate rheomerty)	68
3.4.3. High shear melt rheology (Capillary rheomerty)	70
3.5. Thermal characterization	72
3.5.1. Melting behavior and thermal stability	72
3.5.2. Non-isothermal crystallization kinetics	73

3.5.3. Jeziorny theory (Modified Avrami equation)	79
3.5.4. Ozawa analysis	81
3.5.5. Mo equation	85
3.5.6. Activation energy of crystallization	86
3.6. Summary	88
References	90-91
Chapter-4: Mechanical properties and fracture behavior of PA-612/POE-g-MA blends	92-121
4.1. Overview of the chapter	92
4.2. Dynamic mechanical analysis	92
4.3. Quasi-static mechanical properties	94
4.4. Theoretical modelling of elastic modulus (E) and yield strength (σ_y)	95
4.5. Morphology-toughness correlations	98
4.6. Essential work of fracture measurements	100
4.6.1. Fracture behavior of the blends	100
4.6.2. Correlation of essential work of fracture (w_e) and non-essential work of fracture (βw_p) with domain size (D_n) of blends	102
4.7. Fractured surface morphology	104
4.8. Discussion on toughening of polyamides	106
4.9. Crack resistance behavior and fracture kinetics	110
4.10. Strain field analysis	111
4.11. Optimization of blend	116
4.12. Summary	117

References	120-121
Chapter-5: Morphological, rheological and thermal properties of PA-612/POE-g-MA/HNT ternary nanocomposites	122-151
5.1. Overview of the chapter	122
5.2. Structural characterization of the nanocomposites	122
5.3. Transmission electron microscopy	123
5.4. Cryo-fractured surface morphology and phase distribution	124
5.5. Rheological behavior of nanocomposites	126
5.5.1. Melt flow index (MFI)	126
5.5.2. Low shear melt rheology (Parallel plate rheometry)	126
5.5.3. High shear melt rheology (Capillary rheometry)	129
5.6. Thermal characterization	130
5.6.1. Melting behavior and thermal stability	130
5.6.2. Non-isothermal crystallization kinetics	133
5.6.3. Jeziorny theory (Modified Avrami equation)	139
5.6.4. Ozawa analysis	140
5.6.5. Mo equation	141
5.6.6. Activation energy of crystallization	146
5.7. Summary	148
References	150-151
Chapter-6: Mechanical properties and fracture behavior of PA-612/ POE-g-MA/HNT ternary nanocomposites	152-179
6.1. Overview of the chapter	152

6.2.	Dynamic mechanical analysis	152
6.3.	Mechanical performance	154
6.3.1.	Tensile and flexural properties of nanocomposites	154
6.3.2.	Toughness assessment	156
6.2.3.	Theoretical modeling of mechanical properties	158
6.4.	Essential work of fracture (EWF) measurements	160
6.4.1.	Fracture behavior of the nanocomposites	160
6.4.2.	Correlation of EWF (w_e) and normalized crack propagation resistance ($w_e/\beta w_p$) with domain size (D_n) of nanocomposites	163
6.5.	Fractured surface morphology	168
6.6.	Crack resistance behavior and fracture kinetics	169
6.7.	Strain field analysis: Synchronization of load-time and strain field	171
6.8.	Summary	176
	References	178-179
	Chapter-7: Summary and future scope	180-187
7.1.	Summary of the thesis	180
7.2.	Conclusions	186
7.3.	Future scope of the work	186
	List of abbreviations and symbols	
	List of publications and biography	
	Resume	

List of figures

Figure No.	Title	Page No.
Fig. 1.1:	Notched Izod impact strength as a function of domain size (D_w) for a typical ‘super-tough’ thermoplastic blends	6
Fig. 1.2:	Schematic showing the (a) polymer/platelet clay nanocomposites (b) polymer/HNT nanocomposites	18
Fig. 2.1:	DENT specimen showing fracture process zones	58
Fig.2.2:	Kinetic parameters of a growing crack	61
Fig.2.3:	Test set-up Zwick universal tensile testing machine (Z250) assembled with camera; (1) Aramis-GOM, (2) UTM, (3) grips, (4) structured DENT specimen fixed on grips, (5) teXpert software assisted system and (6) strain field images and multi-stage sections on display	61
Fig. 3.1:	WAXD of PA-612 and PA-612/POE-g-MA blends	65
Fig. 3.2:	SEM micrographs of cryogenically fractured and xylene-etched surfaces of PA-612/POE-g-MA blends: Magnification ratio (X 12,000)	67
Fig. 3.3:	Variation of (a) complex viscosity (η^*), (b) storage modulus (G'), (c) loss tangent ($\tan\delta$) and (d) modified Cole-Cole plots for the PA-612/POE-g-MA blends	70
Fig.3.4:	(a) Shear viscosity as function of shear rate and (b) log shear viscosity as a function of log shear rate	71
Fig. 3.5:	DSC thermograms (a) heating curves, (b) cooling curves, (c)	73

	crystallinity (%) from DSC and XRD and (d) TGA traces of blends	
Fig.3.6:	DSC crystallization curves of PA-612 and PA-612/POE-g-MA blends at various cooling rates	75
Fig.3.7:	Plots of X_t versus T for crystallization of PA-612 and PA-612/POE-g-MA blends at various cooling rates	76
Fig.3.8:	Plots of X_t versus t for crystallization of PA-612 and PA-612/POE-g-MA blends at various cooling rates	77
Fig. 3.9:	Plots of $\log[-\ln(1-X_t)]$ versus $\log t$ for PA-612 and PA-612/POE-g-MA blends	82
Fig. 3.10:	Plots of $\log[-\ln(1-X_t)]$ versus $\log \phi$ for PA-612 and PA-612/POE-g-MA blends at various temperature	83
Fig. 3.11:	Plots of $\log \phi$ versus $\log t$ for PA-612 and PA-612/POE-g-MA blends at different relative crystallinity	84
Fig. 3.12:	Activation energy of crystallization during non-isothermal crystallization for different blend compositions by Takhor method	87
Fig. 4.1:	Dynamic mechanical analysis plots of the blends (a) storage modulus (E'), (b) loss modulus (E'') and (c) loss tangent ($\tan \delta$) versus temperature for the PA-612/POE-g-MA blends	94
Fig. 4.2:	(a) Stress (σ) versus strain (%) of blends and (b) tensile modulus (E), ultimate tensile strength (σ_u), strain-at-break (%) of the blends as a function of POE-g-MA content	95
Fig. 4.3:	Theoretical model-fits for mechanical response (a) elastic modulus (E) and (b) yield strength (σ_y) of the blends	97

Fig. 4.4:	Morphology impact-toughness correlations of PA-612/POE-g-MA blends	99
Fig.4.5:	(a) Self-similarity of load-displacement curves of the investigated blends for various ligament lengths (b) Hill's analysis plot: net section stress versus ligament length (c) variation of specific work of fracture with ligament length for PA-612 and PA-612/POE-g-MA blends	102
Fig.4.6:	Variation of essential work of fracture (w_e) and non-essential work of fracture (βw_p) and impact strength with domain size (D_n) of blends	103
Fig. 4.7:	SEM micrographs of fractured surfaces of PA-612/POE-g-MA blends	105
Fig. 4.8:	Topological schematic of the PA-612/POE-g-MA chain interaction and proposed reaction between PA-612/POE-g-MA	109
Fig. 4.9:	Kinetics of crack growth (a) crack extension (b) CTOD as a function of time and (c) CTOD with crack extension of PA-612 and PA-612/POE-g-MA blends	110
Fig. 4.10:	Load-time-strain field plots of PA-612 and PA-612/POE-g-MA blends	113
Fig. 4.11:	Multi-stage section diagrams of PA-612 and PA-612/POE-g-MA blends	115
Fig. 5.1:	WAXD of nanocomposites	122
Fig. 5.2:	TEM images of nanocomposites	123
Fig. 5.3:	SEM micrographs of cryogenically fractured and xylene-etched surfaces of PA- 612/POE-g-MA and its nanocomposites	125
Fig. 5.4:	Variation of (a) complex viscosity (η^*), (b) storage modulus (G'), (c) loss modulus (G'') and (d) loss tangent ($\tan\delta$) as a function of	127

	frequency and (e) modified Cole-Cole plots for nanocomposites	
Fig.5.5:	(a) Shear viscosity as function of shear rate and (b) log shear viscosity as a function of log shear rate	129
Fig. 5.6:	DSC thermograms (a) heating curves, (b) cooling curves, (c) TGA traces of nanocomposites	131
Fig. 5.7:	DSC crystallization curves of nanocomposites at various cooling rates	135
Fig.5.8:	Plots of X_t versus T for crystallization of nanocomposites at various cooling rates	136
Fig. 5.9:	Plots of X_t versus t for crystallization of nanocomposites at various cooling rates	137
Fig. 5.10:	Plots of $\log[-\ln(1-X_t)]$ versus $\log t$ for nanocomposites	143
Fig. 5.11:	Plots of $\log[-\ln(1-X_t)]$ versus $\log \phi$ for nanocomposites at various temperature	144
Fig. 5.12:	Plots of $\log \phi$ versus $\log t$ for nanocomposites at different relative crystallinity	145
Fig. 5.13:	Activation energy of crystallization during non-isothermal crystallization for different nanocomposites compositions by Takhor method	147
Fig. 6.1:	Dynamic mechanical analysis plots of the blends (a) storage modulus (E'), (b) loss modulus (E'') and (c) loss tangent ($\tan \delta$) versus temperature of nanocomposites	153
Fig. 6.2:	(a) Stress (σ) versus strain (%), (b) strain-at-break (%), (c) tensile modulus (E), ultimate tensile strength (σ_u), (d) flexural modulus,	155

	flexural strength of the nanocomposites as a function of HNT content	
Fig. 6.3:	Variation of Izod impact strength, toughness and brittleness index of nanocomposites	157
Fig. 6.4:	Comparison of experimental data for tensile modulus of nanocomposites with theoretical models	159
Fig. 6.5:	Self-similarity of load-displacement diagrams of the investigated nanocomposites	161
Fig. 6.6:	(a) Hill's analysis plot: net section stress (σ_n) versus ligament length (b) variation of specific work of fracture (w) with ligament length (l) for nanocomposites (c) variation of essential work of fracture (w_e) and non-essential work of fracture (βw_p) as a function of HNT content	162
Fig. 6.7:	Variation of Izod impact strength, essential work of fracture (w_e), crack propagation normalized fracture/resistance ($w_e/\beta w_p$) and entanglement density as a function of HNT content	163
Fig. 6.8:	SEM micrographs of fractured surfaces of nanocomposites	168
Fig. 6.9:	(a) Crack tip opening displacement (CTOD, δ) versus time, (b) crack extension (Δa) versus time (t) plots and (c) δ versus Δa of nanocomposites	170
Fig. 6.10:	Load-time-strain field plots of nanocomposites	172
Fig. 6.11:	Multi-stage section diagrams for nanocomposites	175

List of abbreviations and symbols

ABS	-	Poly(acrylonitrile-co-butadiene-co-styrene)
ASTM	-	American Society for Testing and Materials
AFM	-	Atomic Force Microscopy
BI	-	Brittleness index
CaCO ₃	-	Calcium carbonate
CNT	-	Carbon nanotubes
CTOD	-	Crack-tip opening displacement
D_n	-	Number average dispersed domains size
D_w	-	Weight average dispersed domains size
DENT	-	Double-edge-notched-tension
DIC	-	Digital image correlation
DMA	-	Dynamic mechanical analysis
DSC	-	Differential scanning calorimeter
E	-	Young's modulus
E'	-	Storage modulus
E''	-	Loss modulus
EBR	-	Ethylene-butene rubber
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
EVA	-	Ethylene Vinyl Acetate
EWf	-	Essential work of fracture
FPZ	-	Frontal process zone
G	-	Strain-energy release rate
G'	-	Melt storage modulus
G''	-	Melt loss modulus
G _c	-	Critical strain-energy release rate
HDPE	-	High density polyethylene
HIPS	-	High impact polystyrene
IFPZ	-	Inner fracture process zone
J _c	-	Critical J-integral value
K	-	Stress-intensity factor
KCP	-	Kinetics of crack propagation
K _c	-	Critical stress-intensity factor
K _{IC}	-	Fracture toughness
<i>l</i>	-	Ligament length
LEFM	-	Linear elastic fracture mechanics
LDPE	-	Low density polyethylene
LDPE-g-MA	-	Low density polyethylene grafted maleic anhydride
MFI	-	Melt flow index
MMT	-	Montmorillonite
NBR	-	Butadiene-acrylonitrile rubber

NE	-	PA-612/POE-g-MA
NEH	-	PA-612/POE-g-MA/halloysite
N-EWF	-	Non-essential work of fracture
OPDZ	-	Outer plastic deformation zone
PA	-	Polyamide
PBT	-	Polybutylene terephthalate
PC	-	Polycarbonate
PET	-	Polyethylene terephthalate
PEEK	-	Polyether ether ether ketones
PEGMA	-	Poly(ethylene-co-glycidyl methacrylate)
POE	-	Poly(ethylene -co-octene)
POE-g-MA	-	Poly(ethylene-co-octene) grafted maleic anhydride
PP	-	Polypropylene
PP-g-MA	-	Polypropylene grafted maleic anhydride
PPO	-	Poly(phenylene oxide)
PS	-	Polystyrene
PVC	-	Poly(vinyl chloride)
PVDF	-	Poly(vinylidene fluoride)
PYFM	-	Post-yield fracture mechanics
SAN	-	Styrene-acrylonitrile copolymer
SBR	-	Styrene-butadiene rubber
SEBS	-	Styrene-ethylene-butylene styrene block copolymer
SEBS-g-MA	-	Maleated styrene-ethylene-butylene-styrene

SEM	-	Scanning Electron Microscopy
$\tan\delta$	-	Tangent delta
TEM	-	Transmission electron microscopy
TGA	-	Thermogravimetric analysis
WAXD	-	2D Wide Angle X-ray Diffraction
XRD	-	X-ray diffraction
T_c	-	Crystallization temperature
T_g	-	Glass transition temperature
T_m	-	Melting temperatures
T_p	-	Crystallization peak temperature
$t_{1/2}$	-	Half time of crystallization
δ	-	Crack-tip opening displacement
$d\delta/dt$	-	Crack-tip opening displacement rate
J_c	-	Critical J -integral value
X_c	-	Degree of crystallinity
W_f	-	Total fracture work
w_f	-	Specific total fracture work
w_e	-	Essential work of fracture
βw_p	-	Nonessential work of fracture
W_s	-	Specific wear rate
w_{pp}	-	Weight fraction of PP
β	-	Plastic zone shape factor
σ_y	-	Yield stress

ΔH_f	-	Measured enthalpy of melting
ΔH	-	Ideal enthalpy of melting a perfect crystal
Δa	-	Crack-extension
δ	-	Crack-tip opening displacement
ε_b	-	Elongation-at-break
ρ	-	Density
σ_s	-	Tensile strength
σ_y	-	Yield stress
η^*	-	Complex viscosity