



CHAPTER III EXPERIMENTAL

3.1 Materials

Materials using in this work were given in Table 3.1.

Table 3.1 Summary of materials used in this study.

Material	Grade	Source	Property	Function
LLDPE	Film extrusion (L1810FI)	Thai Polyethylene Co., Ltd.	MFI = 1 (g/10 min) Density = 0.918(g/cm ³) T _m = 137.5 (°C)	Polymer matrix
NR	STR 5L	Rayong Bangkok Rubber	Density = 0.9 (g/cm ³) Equilibrium T _m = 35.5 (°C)	Minor phase
MA	Analytical	Fluka	Density = 0.2 (g/cm ³) T _m = 52.6 (°C)	Forming <i>in situ</i> compatibilizer
DCP	Analytical	Fluka	T _m = 39-41 (°C)	Initiating free radical

All of materials were used as received.

3.2 Methodology

3.2.1 Blending preparation

Brabender Plasti-Corder PL-2000 batch mixer was used to prepare the blends. Blending conditions was shown in Table 3.2.

Table 3.2 Blending conditions.

LLDPE/NR	Temperature (°C)	Rotor speed (rpm)
90/10	150	50
80/20	150	50
50/50	150	30
40/60	150	30

For blending procedure, first NR was masticated on two roll-mill at room temperature for 10 min. After that masticated NR was grafted with 25% MA at room temperature for 5 min by a mixer type N 50. The resultant was MA grafted NR (NR-MA). Then the mixer type N 50 was removed and replaced by the mixer type W 50. When chamber mixer was reached 150 °C, LLDPE was added and molten for 3 min. Then 75% MA was added and after 1 min mixing DCP was added and mixed for another minute. NR-MA was then mixed until torque was constant. Total mixing time was about 10 min. The blend was removed and brought into shredder unit to make granulate. The compositions of the blends were varied as shown in Table 3.3.

Footnote:

Here % means % wt throughout the book.

Table 3.3 Amount of LLDPE/NR/MA/DCP in the blends (g).

LLDPE	NR	MA	DCP
90	10	3	0
			0.5
			1.0
			1.5
90	10	0	0.5
		1	
		5	
		7	
50	50	7	0
			0.5
			1.0
			1.5
50	50	0	0.5
		1	
		3	
		5	

Amount of MA and DCP of composition 80/20 were selected from the proper composition of 90/10 blend while that of composition 40/60 blend was selected from 50/50.

Footnote:

In Table 3.3 reveal that contents of DCP and MA are relatively small compared to polymers and should be expressed in “phr” unit. The meaning of % DCP or % MA used in this thesis means phr of DCP or MA respectively.

3.2.2 Fracture behavior determination

3.2.2.1 *Impact test*

Break energy per unit thickness was obtained from ITR-2000 Impact Tester at ambient conditions following ASTM D 3763. Test specimens were compressed in round shape. The diameter was 12 cm. Load and speed of impactor were 300 kPa and 4 m/s respectively.

3.2.2.2 *Tear test*

Maximum tear strength or tear resistance was measured by Instron 4206 Universal Testing Machine at ambient conditions following ASTM D 1938. The specimens were cut as shown in Figure 3.1. The thickness of the specimens was 1 mm. Test conditions were given in Table 3.4.

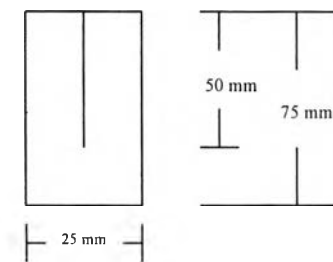


Figure 3.1 Tear test piece.

Table 3.4 Test condition for tear test.

Test parameter	Value
Load cell (kN)	2.5
Initial grip separation (mm)	50
Crosshead speed (mm/min)	50

3.2.2.3 *Tensile test*

Test specimens were brought into Ci3000 Xenon Weather-Ometer following standard method SAE J1885 for 7 days (1 cycle

= 4.8 hr). Weathering condition was shown in Table 3.5 After they were removed from weatherometer, the test was performed by Instron 4206 Universal Testing Machine according to ASTM D 638. Test conditions were summarized in Table 3.6. Dimension of test piece were 6 mm wide, 1 mm thick, and 25 mm gauge length.

Table 3.5 Description of weathering condition for one cycle.

Duration	Time (min)	BPT ^a (°C)	DBT ^b (°C)	RH (%)	Irr (W/m ²)	Fixed blower speed (rpm)	Filter inner/outer
Ligth	228	89	62	50	0.55 @ 340 nm	No	Q/S ^c
Dark	60	38	38	95	0.00	2185	

^a Black Panel Temperature

^b Dry Bulb Temperature

^c Quartz/Type S Borosilicate

Table 3.6 Test condition for tensile test.

Test parameter	Value
Load cell (kN)	5
Initial grip separation (mm)	80
Crosshead speed (mm/min)	100

3.2.2.4 Creep test

Measuring the extension as a function of time at ambient conditions was done by creep tester apparatus according to ASTM D 2990. Test specimens were the same as tensile test. Load 1 kg was used in this test. The extension range or creep was shown in Figure 3.2.

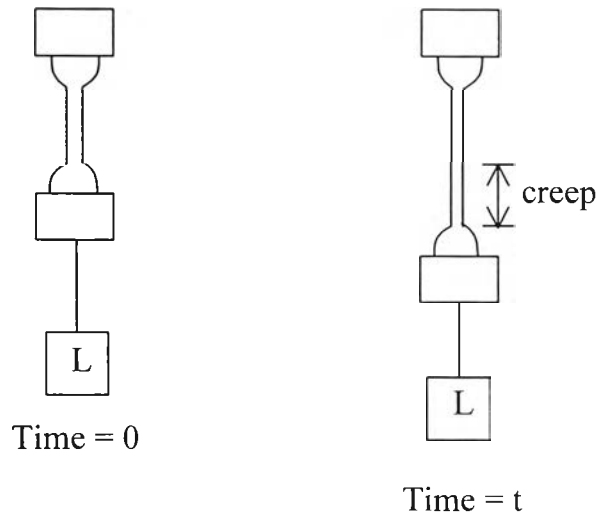


Figure 3.2 Diagram illustrating creep.

3.2.2.5 Fatigue test

Fatigue crack propagation (FCP) analysis was performed by Lloyd Testing Machine in cyclic mode at ambient conditions following ASTM D 671. Single edge crack specimens were used in this test. Test specimens were cut in 25 mm width and 128 mm length. The thickness was 3 mm. A notch 1.5 mm (a_0) was cut with a razor blade. The test specimens were shown in Figure 3.3. Crack length (a) or crack propagation was measured by vernier every 500 cycles. Test conditions were given in Table 3.7.

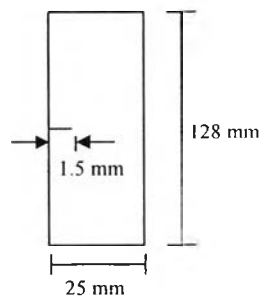


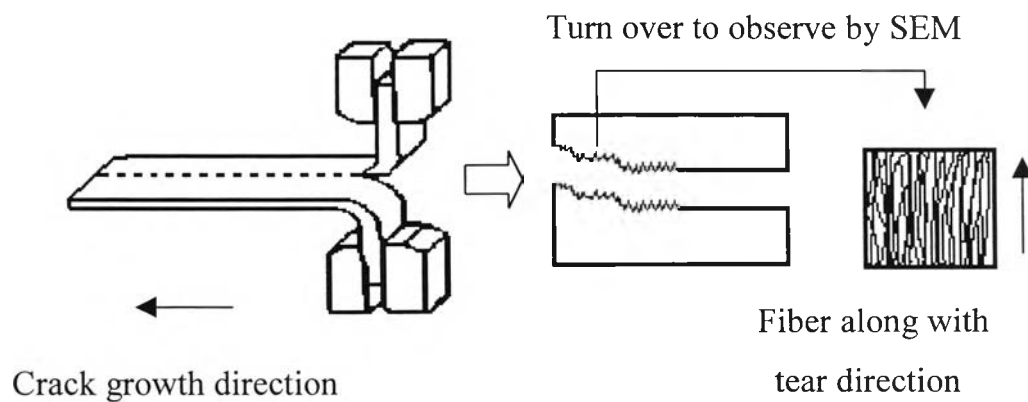
Figure 3.3 Single-edge crack specimen.

Table 3.7 Test condition for fatigue test.

Test parameter	Value
Load cell (kN)	2.5
Frequency (Hz)	0.5
Maximum strain (%)	20
Minimum strain (%)	0

3.2.3 Morphological characterization

Tear-fractured surfaces were coated with gold and observed by a JEOL JSM 6400 scanning electron microscope (SEM) at 15 kV. The magnification was 1000X.

**Figure 3.4** Diagram illustrating SEM crack growth direction.