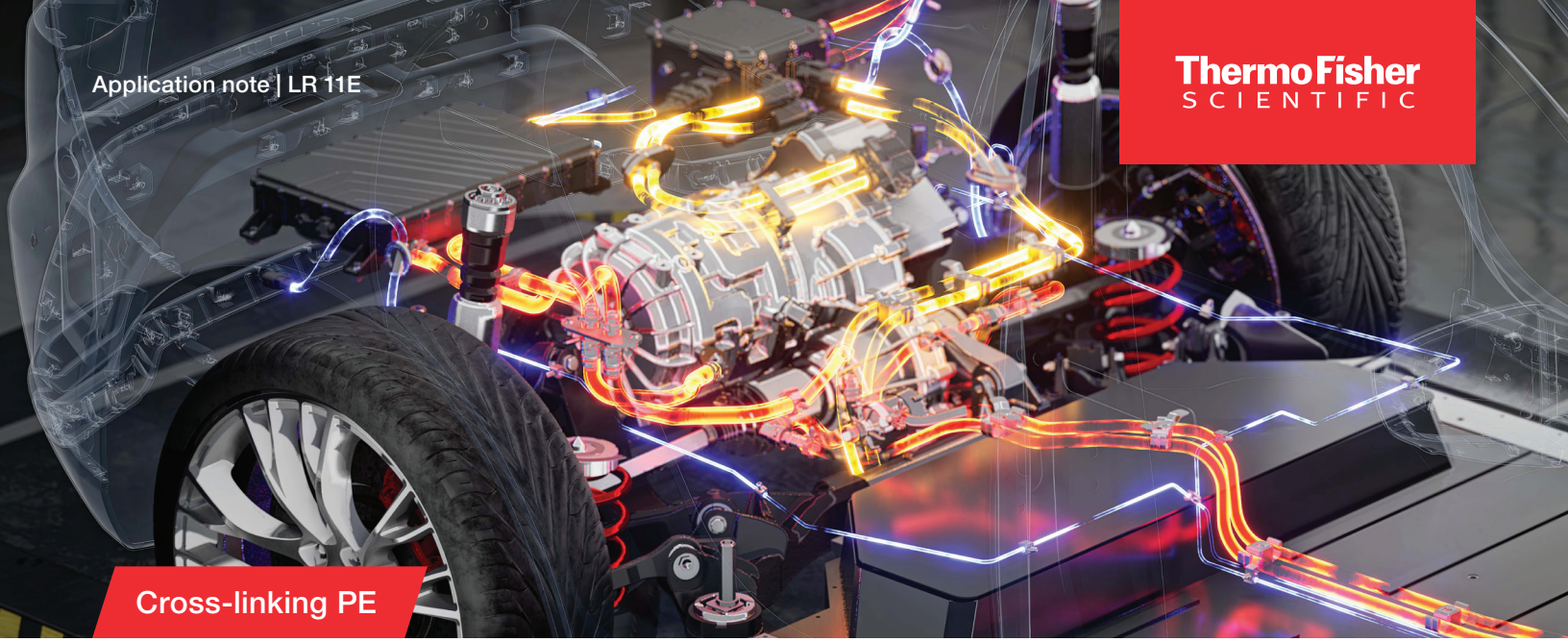

Cross-linking PE

Flow and curing behaviour of cross-linking Polyethylene (XLPE)

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Introduction

Cross-linking PE (XLPE) is widely used in the cable and the automotive industry. XLPE is processed in extrusion and injection moulding process at defined temperatures and then cross-linked at higher temperatures. For this reason, the melting, flow and cross-linking characteristic (processing temperature, cross-linking temperature, cross-linking time, etc.) are of particular interest for the compound development and for the processing parameters.

Test purpose

Determination of the influence of the processing temperature on the cross-linking behaviour of a XLPE sample.

Test equipment

- Torque rheometer system Thermo Scientific™ HAAKE™ PolyLab™ OS Torque Rheometer
- Electrically heated laboratory mixer Themo Scientific™ HAAKE™ Rheomix600 Laboratory Mixer
- Roller rotors
- Thermo Scientific™ HAAKE™ PolySoft Mixer Software

Test conditions

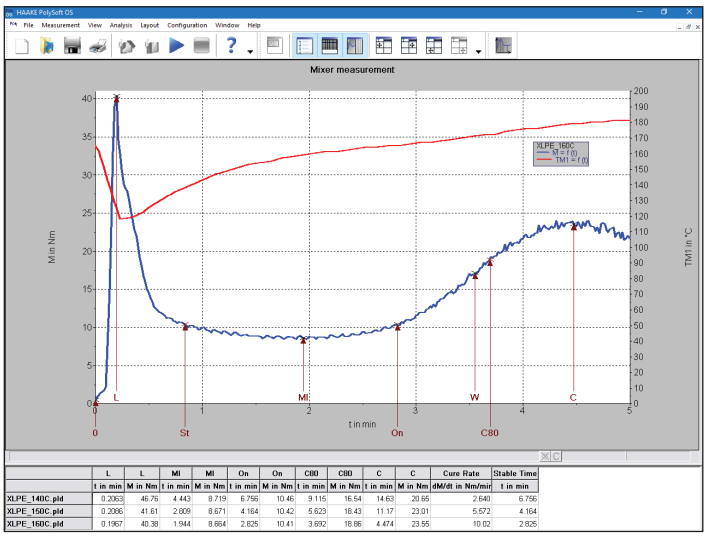
- Rotor speed: 50 rpm
- Sample weight: 45 g
- Mixer temperature:
  a) 140 °C
  b) 150 °C
  c) 160 °C

Test material

- Cross-linking Polyethylene (XLPE)

Test results

Diagram 1 shows the result of the mixer test done with the test sample at a mixer temperature of 160 °C. The graph displays the mixing torque and the sample temperature against the mixing time.



	L	L	MI	MI	On	On	C80	C80	C	C	Cure Rate	Stable Time
	t in min	M in Nm	t in min	M in Nm	t in min	M in Nm	t in min	M in Nm	t in min	M in Nm	dM/dt in Nm/min	t in min
XLPE_140C.pld	0.2053	46.76	4.443	8.719	6.756	10.46	9.115	16.54	14.63	20.65	2.640	6.756
XLPE_150C.pld	0.2086	41.61	2.809	8.671	4.164	10.42	5.623	18.43	11.17	23.01	5.572	4.164
XLPE_160C.pld	0.1967	40.38	1.944	8.664	2.825	10.41	3.692	18.86	4.474	23.55	10.02	2.825

Diagram 1. Result Mixer Test at 160 °C.

At the beginning of the test the cold XLPE-pellets are forced into the running mixer by means of a ram. This is causing a fast increase in torque and results in the first torque maximum, the so-called loading peak ("L"). Due to the high mixer temperature the polymer pellets are melting, and the torque comes down to a minimum ("MI"). The torque value at that point can be used as a relative measure for the melt viscosity.

The beginning of the cross-linking process is causing a second rise of the mixer torque. The point when the torque is reaching a value of 20 % above the torque at the minimum is recorded as the onset of cross-linking ("On"). The torque keeps on rising to a second maximum, the cure maximum ("C"). At this point the cross-linking process has ended, and the sample starts to be grinded in the mixer, the torque is not increasing anymore, and the test is finished.

Points of interest along the torque/time curve:

- **0**: Start point - torque threshold of 0.5 Nm
- **L**: Loading maximum
- **St**: Stable point – 120 % of minimum torque
- **MI**: Minimum torque
- **On**: Onset – 120 % of minimum torque
- **W**: Point of inflection
- **C80**: Point with 80 % of cure maximum
- **C**: Cure maximum

The time difference between the stable point ("St") and the onset of cross-linking ("On") is called the Stable Time. This time difference gives valuable information about the time the material can be processed before it starts to cross-link.

The gradient between "On" and "C80" is called the Cure Rate and gives information on the cross-linking speed.

Diagram 2 shows the torque curves of the three mixer tests in one graph. Clearly it can be seen how an increase in temperature shortens the time before the sample starts to cross-link. Also the cross-linking speed increases with higher temperatures.

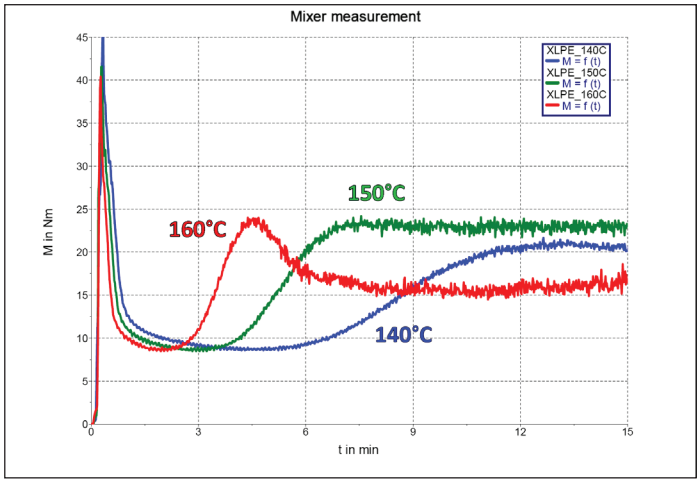


Diagram 2. Comparison Mixer Tests at 140 °C, 150 °C and 160 °C.

Conclusion

These tests show how the laboratory mixer can be utilized as a useful and easy to use tool to investigate the cross-linking and processing behaviour of polymer samples.

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