Dynamic mechanical and impact properties of polypropylene/EPDM blends

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It was established by instrumented impact testing on notched Charpy specimens (DIN 53453 standard, No. 2 bar) that PP homopolymers impact modified by EPDM sorts of different melt viscosities at a rate lower than 10% were subject to brittle fracture in a wide temperature range. The most efficient of the EPDM impact modifiers had melt viscosities similar to that of the starting PP under the conditions of mixing. The course of maximum load at rupture ($F_{\rm max}$) and notched impact strength as functions of temperature showed some analogies with one another as well as with the dynamic mechanical storage (E') and with the mechanical loss factor (tan δ). Thus, linear regression analysis was applied to the following relations: F_{max} vs. (E'; tan δ ; impact strength), F_{max}^2 vs. (tan δ , impact strength) and impact strength vs. tan δ . The optimum correlation coefficients were obtained for $F_{\sf max}$ vs. F and impact strength vs. tan \$. The supposed linearity of the former relation suggested that notched small Charpy specimens behaved as linear elastic bodies at high-rate three-point bending while the latter function referred to the significant role of relaxation of the EPD M impact modifier in the dissipation of impact energy during brittle or semi-brittle fracture of the two-phase PP/EPDM blends. The above relations are rendered probable by the fact that frequency of impact load is 10^2 to 10^3 Hz while that of the dynamic mechanical measurements is about $10¹$ Hz.

Keywords Polypropylene; ethylene/propylene/diene terpolymers; polymer blend; impact strength; dynamic mechanical properties; fracture mechanics

INTRODUCTION

Many recent reports treated the structure-property relationship of'impact modified polypropylene (PP) through its melt blending with ethylene/propylene copolymers (EPM) or ethylene/propylene/diene terpolymer (EPDM) rubbers¹⁻⁹. The impact strength and other physico-mechanical characteristics are the centre of interest as functions of the morphological structure^{1-3,5,6,8,9}. The topic is timely, not only due to the ever-increasing use of impact modified PP instead of HDPE, medium and high impact PS and ABS, but also because of the possibility of tailoring PP properties by incorporation of adeqtaate impact modifiers. In this way, PP rpocessors can be rendered independent of the selection offered by the manufacturers of the polymer.

Definite relations exist between mechanical properties such as impact strength or dynamic mechanical behaviour and molecular structure of polymers 10^{-14} , as was reviewed comprehensively by Boyer¹⁰ and Vincent¹². Gill and Hassel¹⁵ found a correlation between the intensity of loss peak at -110° C measured by dynamic mechanical analysis (d.m.a.) and the drop weight impact strength determined at -29° C for impact modified PP. This connection refers to the importance of the frequency-temperature relationship. In two- or multiphase systems, toughness is usually correlated with the area of the secondary transition peak (the rubbery loss peak is integrated between two temperatures) as proposed by Wagner and Robeson¹⁶ as well as Keskkula *et al.*¹⁷. Sacher¹³ emphasized some relations between the impact strength and the dynamic mechanical dissipation factor, determined under the same temperature and frequency conditions, rather than with the area under the loss peak of rubbery impact modifier. However, Hiltner and Baer¹⁸ and Ramsteiner¹⁹ demonstrated that no quantitative correlation could be expected between the relaxations measured in the region of linear viscoelasticity and the yield and fracture in which highly non-linear effects dominated. Casiraghi and Savadori²⁰ claimed that plastic deformations and molecular relaxation phenomena for a wide variety of polymers would hardly play any part in high-speed impact. On the contrary, Vincent¹² emphasized that pronounced peaks in brittle impact strength of various polymers occurred in the vicinity of their peaks in loss tangent.

The present paper aimed to determine which relations, if any, existed between the maximum load at fracture (F_{max}) or notched impact strength (measured by instrumented impact test DIN 53453 on notched small Charpy specimens) and d.m.a, test characteristics (such as dynamic mechanical dissipation factor (tan δ), complex modulus of elasticity and its components, i.e. the storage (E') and loss (E'') moduli).

Table I **Used materials and their characteristics**

* Prospect **data**

EXPERIMENTAL

The characteristics of materials are summarized in *Table* 1.

Mass- and number-average molecular masses $(M_w$ and M_n , respectively) of PP and EPDM were determined by g.p.c. (Waters Ass.) in 1,2,4-trichlorobenzene at 130°C. For calculations, calibration curves of PP and PE were used, respectively.

Phase viscosity ratio (μ) i.e. the ratio of melt viscosity of the dispersed phase (EPDM in this case) to that of the matrix-forming polymer (PP) under the same conditions was determined by measuring Brabender torque values of both pure components at 190 $^{\circ}$ C after 5 min of mixing⁷⁻⁸ *(Table 1).* The tendency of the phase viscosity ratio is analogous with that of the Mooney viscosity *(Table 1).*

Ethylene content of EPDM-containing ethylidenenorbornene termonomer was determined by i.r. spectroscopy (Perkin-Elmer 577 spectrophotometer) measuring the $A_{1380 \text{ cm}^{-1}}$ to $A_{1460 \text{ cm}^{-1}}$ ratio according to the evaluation method of Corish and Tunnicliffe²¹ using film samples evaporated dry from xylene solution.

Granules of equal size were introduced into a W 50 type kneader chamber of a Brabender PLV 151 Plasticorder both for PP and the EPDM additive. Mixing was conducted at 30 rpm for 5 min at 190°C. Specimens were prepared from the compression moulded sheets of blend. 150×150 mm sheets of 1 or 4 mm in thickness were compression moulded between flat mould halves after a 4 min preheating and a 2 min precompression at $5-10\%$ of the final pressure of 10 MPa at 190°C. Compression moulding was immediately followed by an undercooling treatment at 50°C for 30 min resulting in the desired homogeneous microstructure. The appropriate morphology was then checked by polarization microscopy. Finally 50×8 mm prisms were cut from the 4 mm sheets which were milled by a Göttfert Fras-boy mill into the standard (DIN 53453) specimens. A U-shaped notch of 0.8 mm in width was placed at one-third of the thickness of each specimen by a Ceast slot-cutter.

Notched impact strengths were measured by a Ceast AFS/MK-2 fractoscope at loads between 3.82 and 9.55 daN using a hammer of 0.29 kg (2J) at a dropping angle of 150° and dropping speed of 3.70 m s⁻¹. Sweep time was 2-32 ms, adjustment of pretrigger and trigger was 1/4. Specimens with slots were put into a glass beaker which was immersed into the thermostating liquid.

The fractoscope recorded load vs. time and displacement vs. time plots. Impact strengths were determined from 5-7-fold runs. In order to examine the temperature dependence of impact strength, sub-ambient measurements were also made in an ethanol/dry ice bath, varying the temperature by the amount of dry ice.

Mechanical loss tangents and elasticity moduli were measured by Du Pont Series 990 Thermal Analysis System as functions of temperature, using its 980 Type Dynamic Mechanical Analyzer. The compression moulded 1 mm thick sheets were machined into specimens. Measurements were carried out under N_2 from -150° to $+150^\circ$ C at 5° C min⁻¹ heating rate. Oscillation amplitude was 2.0 mm, A/Z gain 20% , while sensitivity was adjusted to 20 or 10 mV cm⁻¹ in the frequency or damping region, respectively. Frequency and damping were recorded against temperature. These plots were processed by a computer for determination of components of the complex modulus of elasticity, i.e. the storage (E') and loss (E'') moduli as well as tan δ values.

RESULTS AND DISCUSSION

Typical load vs. time fractograms recorded in the instrumented Charpy impact test are shown in *Figure 1* as a function of temperature for PP impact modified by 5% of Buna AP 447. Since the fractograms are taken in linear displacement (deflection) vs. time mode, the areas under load vs. time curves are proportional to the notched impact strengths of the specimens. Specifically, the load vs. time curves are characteristic to brittle and semi-brittle fracture when recorded below 40°C and at 60°C, respectively. In the latter case, a short horizontal section is observed around F_{max} referring to the occurrence of some plastic deformation process.

Appearance of a brittle to ductile transition depends not only on the frequency of impact load and temperature but also on the composition of the binary system and its structure and morphology. Asar *et al. 6* concluded from instrumented Izod impact testing of injection moulded PP bars impact modified by EPDM that the ductile to brittle transition is at about room temperature, at a deformation rate of $1.3-1.4$ m s⁻¹. In the present experiments, higher rates (3.7 m s⁻¹) are used; the plastic deformation is still clearly appreciable for specimens of low phase viscosity ratio, i.e. modified by 10% of EPDM

Time (ms)

Figure I **Load vs. time fractograms recorded** in instrumented **impact testing of notched Charpy specimens in PP blended** with 5% of Buna AP 447 at **different temperatures**

Time (ms)

Figure 2 **Load vs, time fractograms recorded in instrumented impact tasting of notched Charpy specimens of blends containing** 10% **of EPDM additives of different melt viscosities (i.e. phase viscosity ratios) at 23°C**

with a melt viscosity close to that of PP (cf. fractograms of notched Charpy specimens at 23°C containing 10% of Buna AP 147 and Buna AP 251 in *Figure 2).* On the contrary, the brittle fracture is characteristic to blend impact modified by EPDM of higher μ value *(Figure 2).*

As can be seen further from *Figure 1,* notched small Charpy specimens behave as linear elastic bodies since their stiffness increases with decreasing temperature (i.e. the initial slope of load vs. time curves is decreasing monotonically with increasing temperature). This was also observed by Casiraghi²² for razor-notched PBTPbased Charpy samples in three-point bending test at high rate. He found it satisfactory to apply the principle of linear elastic fracture mechanics *(LEFM)* for calculation of the corresponding characteristics $(K_c$ and G_c).

By comparison of the areas under curves of *Figure 2,* it can be established that the highest increases in impact strength are achieved with EPDM types (Buna AP 147 and Buna AP 251) having melt viscosity close to that of the PP matrix¹ i.e. the value of phase viscosity ratio is near to unity 8'9. Degree of dispersity of the elastomeric impact modifier in the PP matrix is mainly determined by the melt viscosity difference between the substrate to be modified and the impact modifier under the conditions of melt blending^{4, 7}. E.g. PP samples containing 10% of Buna AP 147, 251, and 341 showed average particle size of the dispersed EPDM elastomer between 0.55 and 0.60 μ m while the blends prepared with other EPDM types comprised particles between 0.64 and 0.90 μ m^{7,8}.

Maximum forces (F_{max}) read on load vs. time fractograms-reached usually at fracture due to the brittle or semi-brittle samples-and dynamic storage moduli (E') derived from the d.m.a. spectra are plotted in *Figure 3* against the corresponding temperatures at which the three-point impact bending tests were conducted.

It can be stated on the basis of *Figure 3* that the course of F_{max} and E' is similar in temperature. A maximum or shoulder in F_{max} vs. temperature curves appears in the

Figure 3 Maximum load (F_{max}) in instrumented impact testing **of notched specimens of blends containing** 5% (©) and 10% (e) Buna **AP 447 as well as dynamic storage modulus (E') read** from d.m.a, **spectra as functions of temperature**

Figure 4 '=max values measured by instrumented Charpy **impact** tests for blends containing 5% (^O) and 10% (^O) of Buna AP 447 EPDM at different **temperatures plotted against** E' modulus **reed** from the d.m.e, spectre at corresponding temperatures

Figure 5 Notched impact strength and **mechanical loss factor** $(\tan \delta)$ of PP blends impact modified by 5% \textcircled{c} and 10% \textcircled{e} of Buna AP 251 **as functions of** temperature

vicinity of the correspond transitions of the PP/EPDM two-phase system. T_g or the β -transition of PP homopolymer is at about room temperature while that of EPDM is at -40° C (the more pronounced β -transitions are shown in *Figures 5* and 6). The corresponding F_{max} and E' values are plotted in *Figure 4*. It can be seen that F_{max} vs.

 E' points are well approximated by a straight line. This relationship supports the conclusion for the linear elastic behaviour of the samples drawn from *Figure 1.*

An interesting result is derived by consideration of curves in *Figures 5* and 6, where notched impact strengths and mechanical loss factors (tan δ) are plotted, respectively, against the temperature of impact testing.

By comparison of cruves in *Figures 5* and 6, one can see that the tendency of notched impact strength is analogous with that of tan δ in temperature, i.e. a maximum and a shoulder are observed between -40° and -20° C and around room temperature, respectively. Thus, a linear relationship can be assumed between the notched impact strength and tan δ determined at different temperatures.

Comparing *Fioure 3* with *Figures 5* and 6, a linear relation is also suggested between the notched impact strength or F_{max} and the mechanical loss factor (tan δ).

In a previous paper 9, the *LEFM* theory was assumed to be valid for the results of instrumented impact tests performed on standardized small Charpy bars with a Ushaped notch and also a linear relation was proposed between fracture toughness or critical stress intensity factor (K_c) and mechanical loss factor as well as between the fracture energy or the critical strain energy release rate (G_c) and the mechanical loss factor. Since K_c is directly proportional to F_{max} while $G_c \propto F_{\text{max}}^2$ (or more precisely, to F_{max}^2 /*E*), the regression treatment was applied also to pairs of values including F_{max}^2 of F_{max}^2/E' . The supposed linear relationship between F_{max}^2/E' and tan δ is shown in *Figure* 7 for blends containing 5 and 10% of Buna AP 447.

Since the dynamic mechanical properties were determined at $10¹$ Hz frequency range while the notched Charpy specimens were fractured at a high speed, i.e. the frequency of impact bending was in the range of 3×10^2 to $10³$ Hz as deduced from the rupture time which is regarded as an acceptable approximation²³, it should be emphasized that the linear regressions of the above factors in pairs are also considered as approximations.

Figure 6 **Notched impact strength and mechanical loss** factor (tan 8) of PP blends **impact modified** by 5% (O) and 10% (O) **of** Buna AP 447 **plotted against** temperature

Figure 7 F_{max}^2 /*E'* vs. tan δ relation as determined for PP blends containing 5% (O) and 10% (0) of Buna AP 447

All the fractoscopic (F_{max} and notched impact strength) and dynamic mechanical analytical $(E'$ and tan $\delta)$ results are summarized in *Table 2* measured at different temperatures.

According to the above considerations, a linear regression treatment was applied for the following pairs of values:

 $F_{\text{max}}-E'$ F_{max} -tan δ F_{max} -impact strength F_{max}^2 -tan δ F_{max}^2 -impact strength F_{max}^2/E' -tan δ F_{max}^2/E' -impact strength

impact strength-tan 6

The results of linear regression analyses are collected in *Table 3.*

Table 2 Fmax, notched impact strength, and dynamic mechanical properties (E' **and tan** 8) of the studied PP/EPDM blends as functions of temperature and blend composition

Materials composition	Tem- perature $(^{\circ}C)$	F_{max} (N)	Notched impact strength $(kJ m-2)$	E' (GPa)	10^2 x tan δ	Materials composition	Tem- perature $(^{\circ}C)$	F_{max} (N)	Notched impact strength (kJ m^{-2})	ϵ' (GPa)	10^2 x tan δ
5% of Buna	-60	37.3	4.7	3.62	2.95	5% of Buna	-60	32.5	3.7	5.08	1.73
AP 147	-40	31.2	4.3	3.39	3.62	AP 451	-40	31.4	3.9	4.71	2.39
95% of Tipplen	-20	33.0	4.5	3.11	3.94	95% of Tipplen	-20	32.0	3.6	4.28	2.58
H523	$\mathbf 0$	38.2	5.2	2.78	4.57	H523	$\mathbf 0$	31.5	4.8	3.67	3.72
	23	34.0	6.8	1.83	7.82		23	24.6	5.3	2.28	7.58
	40	22.2	6.0	1.39	5.51		40	22.0	5.9	1.71	5.50
	60	22.0	12.0	1.01	5.44		60	18.4	5.8	1.14	6.29
10% of Buna	-60	39.5	4.3	3.07	1.56	10% of Buna	-60	33.3	3.5	3.72	4.26
AP 147	-40	39.4	5.0	2.82	2.32	AP 451	-40	36.8	5.0	3.42	5.03
90% of Tipplen	-20	37.2	4.9	2.48	3.07	90% of Tipplen -20		37.0	5.0	3.19	4.71
H523	0	36.3	6.9	2.13	3.14	H523	0	31.4	6.1	2.86	5.46
	23	35.0	9.0	1.32	7.26		23	27.0	7.3	2.02	8.95
	40	30.3	11.2	0.94	5.65		40	26.2	9,0	1.52	6.71
	60	20.8	no break	0.58	7.31		60	21.9	9.5	1.12	5.92
5% of Buna	-60	33.1	3.7	4.97	1.60	5% of Buna	-60	34.9	3.6	4.44	2.64
AP 447	-40	33.5	3.7	4.74	1.93	AP 341	-40	33.0	3.9	4.05	3.58
95% of Tipplen - 20		35.4	3.9	4.30	2.49	95% of Tipplen	-20	33.6	3.5	3.68	3.81
H523	0	34.2	4.4	3.81	2.93	H523	0	31.9	4.8	3.33	4,59
	23	29.7	4.8	2.57	6.43		23	31.7	7.0	2.21	8.46
	40	25.1	4.9	1.91	4.83		40	24.0	6.7	1.62	6.95
	60	19.2	5.7	1.29	4,98		60	21.9	7.0	1.14	7.02
10% of Buna	-60	31.0	3.9	4.36	1.53	10% of Buna	-60	32,4	3.8	4.00	3.13
AP 447	-40	29.3	4.6	4.02	2.07	AP 341	-40	35.5	4,5	3.54	3.82
90% of Tipplen	-20	30.5	4.9	3.64	2,40	90% of Tipplen	-20	37,8	4.7	3.29	4.08
H523	0	30.1	5.2	3.27	2.67	H523	\mathbf{o}	36.9	6.2	2.88	5,21
	23	25.5	6.4	2.19	6.59		23	28.0	7.2	1.96	8.16
	40	23.8	8.3	1.55	4.96		40	24.6	8.1	1.49	6.80
	60	17.0	9.3	1.06	5.10		60	19.6	8,9	1.10	6.70
5% of Buna	-60	33.0	3.3	4.14	1.96	5% of Buna	-60	33.8	4.1	4.12	2.76
AP 251	-40	37.0	3.7	3.67	2.66	AP 541	-40	33.1	4.8	3.76	3.71
95% of Tipplen	-20	34.8	3.6	3.35	2.98	95% of Tipplen	-20	34.0	4.1	3.47	3.95
H523	0	34.0	5.4	2.93	3.62	H523	0	36.4	5.8	3.13	4.55
	23	34.8	8.2	1.89	7.39		23	32.0	6.7	2.23	8.14
	40	31.3	8.0	1.37	6.02		40	23.7	6.5	1.67	7.36
	60	24.0	8.8	0.97	6.35		60	20.1	7.5	1.23	6.20
10% of Buna	-60	31.9	3.9	3.81	2.61	10% of Buna	-60	32.5	4.9	3.92	2,51
AP 251	-40	36.0	4.1	3.53	3.97	AP 541	-40	34.0	4.4	3.50	3.55
90% of Tipplen -20		37.7	6.2	3.20	3.73	90% of Tipplen	-20	30.0	5,0	3.22	3.38
H523	0	35.1	7.5	2.90	4.49	H523	0	31.6	5.9	2.84	4.20
	23	32.5	11.3	1.98	8.41		23	28.2	8,0	1.93	7.98
	40	28.7	10.9	1.47	6.17		40	24.0	9.9	1.44	6.42
	60	21.0	19.1	1.01	6.37		60	19.8	9,7	1.03	6.65

		Function			$Y = A + B$ X equation		
Y	(unit)	x	(unit)	Correlation coefficient R	A	Β	
F_{max}	$(10^{-1} N)$	E'	$(10^{-9} m^{-2})$	0.692	2.121	0.332	
F_{max} F_{max}^2 F_{max}^2 / E'	$(10-1 N)$ $(10^{-2} N^2)$ $(10^7 N m^2)$	tan δ tan δ tan δ	$(102$ tan $\delta)$ $(102$ tan $\delta)$ $(102$ tan $\delta)$	-0.513 -0.518 0.439	3.742 13.648 2.341	-0.149 -0.865 0.325	
$\frac{F_{\text{max}}}{F_{\text{max}}^2}$	$(10-1 N)$ $(10-2 N^2)$ $(10^7 N m^2)$	imp. str. imp. str. imp. str.	(kJ m^{-2}) $(kJ m-2)$ (kJ m ^{-2})	-0.537 -0.526 0.542	3.764 13.650 2,008	-0.118 -0.664 0.303	
imp.str.	$(kJ m-2)$	tan δ	$(102$ tan $\delta)$	0.639	2.173	0.845	

Table 3 Results of the linear regression calculations

The critical correlation coefficient for 84 measured pairs of values, i.e. for 82 degrees of freedom, was $R_{\text{crit}} = 0.353$ at 99.9% confidence level²⁴. Since the values of *Table 3* are above this R_{crit} level, the studied relations can be ragarded as linear. It should be emphasized, however, that $R > R_{\text{crit}}$ is a necessary but not sufficient condition of linearity. Considering the correlation coefficient values of *Table 3,* the best correlation was obtained for $F_{\text{max}}-E'$ and impact strength-tan δ . According to the former relation, increase of F_{max} involves that of E' as evidence of the linear elastic behaviour of notched small Charpy specimens in the case of brittle fracture. Vincent¹² reported that brittle impact resistance existed for plastics when $E' > 4.49$ GPa. In the present experiments, however, there are some $F_{\text{max}}-E'$ points with E' of 1 GPa where the course of the fractogram can be classified rather as semi-ductile fracture.

According to the notched impact strength-tan δ relation, the increase in brittle impact strength depends on the increment, i.e. identical course of the mechanical loss factor. It suggests that the relaxation phenomena of the rubbery impact modifier and those of PP matrix play important roles in the brittle fracture of PP/EPDM blends. It coincides with the statement of $Kausch²⁵$ that impact resistance of plastics is enhanced by any molecular processes which promote the distribution and dissipation of the impact energy and cause large deflection or displacement before inception of rapid crack propagation.

CONCLUSION

It was established by the DIN 53453 standard instrumented impact tests on Charpy specimens No. 2 of PP homopolymers impact modified by 5 and 10% of EPDM, that impact strength of the blend is increasing as the phase viscosity ratio of the incorporated EPDM is reduced, i.e. the melt viscosity of the latter should be as close to that of the PP homopolymer as possible under the conditions of melt blending. The brittle to ductile fracture transition depends not only on the rate of deformation and the testing temperature but also on the concentration of the elastomer (at low phase viscosity ratios, fractograms of systems containing 10% of impact modifier show plastic deformation even at room temperature, while at 5% EPDM level this feature of load vs. time curves is revealed only at higher temperatures) and on the phase viscosity ration of EPDM and, consequently, on the phase structure of the produced two-phase system. At high

dispersity of the elastomer, large deflection or displacement is attained and thus ductile fracture becomes dominant.

Since the maximum load at fracture (F_{max}) and the dynamic mechanical storage modulus (E') follow a similar course in temperature during instrumented impact testing of the samples, a linear relationship was supposed between them. Plotting F_{max} or notched impact strengths against temperature, in the vicinity of T_a (β -transition) values of the EPDM impact modifier and PP homopolymer (appearing separately in the d.m.a. spectrum), a peak or shoulder is observed. Thus a linear relation was supposed again between F_{max} and the mechanical loss factor (tan δ) as well as between the notched impact strength and tan δ . Linearity of F_{max} vs. $\tan\delta$ or F_{max}^2/E' vs. $\tan\delta$ suggests that fracture mechanical characteristics $(K_c \text{ and } G_c)$ determined by three-point bending of notched Charpy specimens at high rate at different temperatures using the *LEFM* theory show similar dependence on tan δ values of PP/EPDM blends at corresponding temperatures. The linear regression treatment of 84 measured points resulted in quite favourable correlation coefficients for $F_{\text{max}}-E'$ and notched impact strength-tan δ relations. The former function refers to the brittle fracture and linear elastic behaviour of notched Charpy specimens at high impact rate while, according to the latter one, the secondary transitions play a considerable part in the dissipation of impact energy in the brittle fracture of two-phase PP/EPDM blends. Correlation between the results of Chapry impact tests and the dynamic mechanical characteristics makes it probable that the frequency of the former lies between 10^2 and 10^3 Hz whereas that of the latter is about 10^1 Hz.

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