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WORKING PARTY ON STRUCTURE AND PROPERTIES OF COMMERCIAL POLYMERS*

IMPACT TESTING OF POLYPROPYLENE MOULDINGS

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Impact testing of polypropylene mouldings

ABSTRACT: Nine different laboratories have collaborated in a research programme on factors affecting the fracture resistance of injection moulded polypropylene plaques, as measured in the dart impact test. The principal factors investigated were the presence of weld lines and ejector pin marks, and the fracture surface energy $G_{\rm LC}$ of the polypropylene. A comparison was made between a homopolymer and a copolymer (rubber-toughened) grade. It was found that the copolymer had the higher fracture surface energy at 23°C but gave a lower $G_{\rm LC}$ than the homopolymer at -40°C, where the extent of crack-tip yielding was reduced. The weld line constituted an important defect which lowered the dart impact strength, especially when the weld was formed by melt fronts meeting head-on. However, there was no detectable effect of the weld in notched specimens. It is concluded that weld lines act essentially as surface defects. Ejector pin scars, which clearly are surface defects, also significantly reduce dart-impact strength.

INTRODUCTION

This paper is the second in a series concerned with the impact behaviour of thermoplastics mouldings. An earlier study by the IUPAC Working Party on "Structure and Properties of Commercial Mouldings" (1) showed that falling dart and driven dart impact tests detected weaknesses at or near weld lines in moulded polypropylene homopolymer, whereas other tests, including tensile impact, notched Izod and Charpy, and fracture mechanics measurements, were insensitive to the presence of a weld in a moulded item. It was suggested that the dart impact tests were more sensitive because the test conditions coincided with a tough-brittle transition in polypropylene and that the other tests would show the effect of the weld at a higher temperature, at which the transition would be observed in notched specimens. An alternative possibility is that the weld line is basically a surface defect and that the material lying below the surface zone in the weld region is little different from that in the remainder of the moulding. This would explain why notched impact tests fail to detect any weakness at the weld, whereas unnotched tests are sensitive. According to this explanation, the weld constitutes a Griffith flaw at the surface of the moulding.

In the present programme, the fracture properties of polypropylene homopolymer and copolymer are compared. Both conventional notched impact tests and experience under service conditions show that the copolymer is significantly tougher than the homopolymer and it is important to establish the degree to which the two types of fracture resistance can be correlated.

The following abbreviations are used in the text to identify participants in the programme:

BP	BP Chemicals, Barry and Grangemouth U.K.
BW	Borg Warner Chemicals, Amsterdam, Netherlands.
CIT	Cranfield Institute of Technology, Bedford, U.K.
Hoechst	Hoechst AG, Frankfurt, Germany.
ICI	ICI plc, Welwyn Garden City and Wilton, U.K.
Monsanto	Monsanto, Louvain-la-Neuve, Belgium.
MP	Montepolimeri, Bollate, Italy.
RP	Rhone Poulenc, Aubervilliers, France.
TNO	TNO, Delft, Netherlands.

MATERIALS AND MOULDINGS

The two polymers used for this study were Moplen T30S polypropylene homopolymer, supplied by MP, and Propathene GMM101 polypropylene copolymer, supplied by ICI. In both cases, a single batch of material was reserved for the work and supplied to all participants. The homopolymer was of the same grade of material as that used for the previous study by the Working Party, which was mentioned in the Introduction.





Double gated plaques approximately 3.7 mm thick were injection moulded by RP. As shown in Figure 1, the mould produces two types of plaque, one of which is end-gated, so that the flow fronts meet head-on (Type I), the other being side-gated, so that the converging flow fronts travel approximately in the same direction into the mould (Type II). Weld lines are visible on both types of moulding where the melt fronts meet. Moulding conditions were as follows:

	Homopolymer	Copolymer
Barrel temperatures (°C)	230,230,220,225,210 N*	245,237,237,240,225 N*
Plasticisation (s)	40	37
Screw Speed (rpm)	70	70
Pressure (bar)	20	20
Injection	6 s at 70 bar then 37 s at 170 bar	6 s at 70 bar then 170 s at 170 bar
Cooling	25 s at 20 - 46°C	20 s at 20 - 46°C

N indicates position of nozzle.

Figures 2 and 3 illustrate two other types of mould designed and used by ICI to produce mouldings containing weld lines. The first is a multi-cavity Charpy mould in which flow is across the length of the Charpy bar in two cavities and along its length in the other two. Two



Fig. 2 The ICI multi-cavity mould, showing the labelling of positions. Bars are identical in size. Double lines show locations of moulded in notches on upper face.



Fig. 3 The ICI picture frame mould, showing positions from which Charpy bars were cut by BP.

of the bars contain weld lines, one lying along the length of the bar and the other across the bar. Mouldings were made from the copolymer only, both with and without a moulded-in notch on the upper face. The second type of mould is a 'picture frame' which is gated so that the weld line is at the centre of one side of the frame.

Both BP and Hoechst compression moulded 6 mm sheets at $190 \,^{\circ}C$; TNO compression moulded 3 mm sheets at $230 \,^{\circ}C$. Cooling was controlled at $3 \,^{\circ}C$ per minute by BP and at $7 \,^{\circ}C$ per minute by TNO.

MATERIALS PROPERTIES

Measurements of density, yield stress, elongation at break and fracture toughness were made on both polymers. Density measurements were made at 23°C over a period of 130 days after moulding. The other properties were measured over a range of temperatures down to -70°C.

Density

Density changes reflect changes in the structure of the polypropylene which can have a significant effect on mechanical properties. Results presented in Fig. 4 show that the density of compression mouldings increases linearly with log (time), the curves for the two polymers being approximately parallel. Copolymer specimens from the ICI multicavity mould had a much lower density than the corresponding compression mouldings.



Fig. 4 Changes in density with time after moulding. Data of: (□, ■) BP; (o, •) Hoechst; (△, ▲, ▼) TNO.

Yield stress and elongation

Low speed tensile tests were carried out by MP, on ASIM D1822S specimens machined from the RP double gated plaques at a crosshead speed of 16.7 μ /s and an initial grip separation of 25.4 mm. Three specimens were tested for each condition. Results are given in Fig. 5. There was no significant difference in yield stress between the two types of plaque, or between specimens cut along and across the flow direction. However, elongations at break were higher in specimens cut parallel to the flow direction. As expected, the copolymer had a lower yield stress and a higher elongation at break than the homopolymer over the entire temperature range, the differences being particularly marked between -40° and -20°C, where the elastomeric phase of the copolymer is above its glass transition temperature but the polypropylene matrix is below its Tg.

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COPOLYMER

Fig. 5 Yield stress and elongation at break at low strain rate: along (o) and across (●) flow direction. Data of MP.

Fracture toughness in impact

Fracture toughness measurements were made by MP between -80° and $+40^{\circ}$ C, using an instrumented pendulum with an impact velocity of 1 m/s to fracture double edge notched (DEN) specimens in tension. The specimens, in the form of 3.8 x 15 x 60 mm rectangular bars, were machined from RP plaques both parallel to and across the flow direction. Grip separation L was 40 mm and 2a/W was 0.5 in all cases. Notches of length a were made by pressing a fresh razor blade to a depth of 0.1 to 0.2 mm into the end of a saw cut.

Force-deflection curves at low speeds showed non-linearity even at -70° C, so that it was not possible to obtain valid K_{TC} data. By contrast, the correlations were linear in impact tests below -20° C.

Results presented in Table 1 include some data obtained at higher temperatures, where some degree of non-linearity and, therefore plasticity, is evident. The results consist of: (a) short-time values for Young's modulus E, obtained by rebound measurements on unnotched Charpy bars, using the same pendulum, as described in reference (2); (b) K_{IC} values calculated using geometrical Y factors as listed in reference (3); (c) G_{IC} values obtained from K_{IC} /E; and (d) G_{IC} values calculated from the energy U absorbed at fracture and the rate of change of compliance, using the equation:

 $G_{IC} = U/BW\emptyset$ where $\emptyset = \frac{\int Y^2(2a/W)d(2a/W)}{Y^2(2a/W)} + L/(2aY^2)$

The table shows that $K_{1,c}$ exhibits only a small variation with temperature and at -40 C and below is lower in the copolymer than in the homopolymer, an effect also reported by Fernando and Williams (4).

TABLE 1. Properties of polypropylene determined in tensile impact on notched and DEN specimens cut from RP plaques with tensile direction along (A) and across (X) flow direction. * indicates some non-linearity in force-deflection curves. Data of MP.

		Temp	eratur	≘ (^o C)		
	23	0	-20	-40	-60	-80
	POL	YPROPYL	ENE HON	OPOLYM	ER	
A	2.02	3.10	3.70	4.55	4.70	4.96
A X	1.82* 1.79*	1.87* 1.87*	1.83 1.74	1.82	1.96	2.04
A X	1.6 4* 1.58*	1.13* 1.13	0.90 0.82	0.73	0.82	0.84
A X	1.82* 1.75	1.22* 1.25	1.06 1.00	0.78	0.84	0.85
	P	OLYPROP	YLENE C	OPOLYME	R	
A	1.60	2.39	2.84	4.17	4.38	4.68
A X		2.10* 1.75	1.70 1.67	1.59	1.74	1.78
A X		1.69* 1.28	1.02 0.98	0.60	0.69	0.68
A X		1.71* 1.28	1.12 0.98	0.59	0.71	0.70
	A A X A X A X A X A X A X	23 POI A 2.02 A 1.82* X 1.79* A 1.64* X 1.58* A 1.82* X 1.75 P A 1.60 A X A X A X	Z3 O 23 O POLYPROPYL A A 2.02 3.10 A 1.82* 1.87* X 1.79* 1.87* A 1.64* 1.13* X 1.58* 1.13 A 1.82* 1.22* X 1.75 1.25 POLYPROPY A 1.60 2.39 A 2.10* X X 1.75 1.28 A 1.69* X X 1.28 A	Temperature 23 0 -20 POLYPROPYLENE HON A 2.02 3.10 3.70 A 1.82* 1.87* 1.63 X 1.79* 1.87* 1.74 A 1.64* 1.13* 0.90 X 1.58* 1.13 0.82 A 1.64* 1.25* 1.00 X 1.75 1.25 1.00 POLYPROPYLENE C A 1.60 2.39 2.84 A 2.10* 1.70 X X 1.75 1.67 A A 1.69* 1.02 X X 1.28 0.98 A	Temperature (°C) 23 0 -20 -40 POLYPROPYLENE HOMOPOLYM A 2.02 3.10 3.70 4.55 A 1.82* 1.87* 1.83 1.82 X 1.79* 1.87* 1.74 A 1.64* 1.13* 0.90 0.73 X 1.58* 1.13 0.82 0.78 A 1.64* 1.25* 1.00 0.78 X 1.75 1.25* 1.00 0.78 X 1.75 1.25* 1.00 0.78 X 1.75* 1.25* 1.00 0.78 X 1.75* 1.25* 1.00 0.78 X 1.75* 1.67 1.59 X A 1.60 2.39 2.84 4.17 A 2.10* 1.70 1.59 X X 1.28 0.98 4.17 4.1.69* A 1.69*<	Temperature (°C) 23 0 -20 -40 -60 POLYPROPYLENE HOMOPOLYMER A 2.02 3.10 3.70 4.55 4.70 A 1.82* 1.67* 1.83 1.82 1.96 X 1.79* 1.87* 1.74

At higher temperatures, the copolymer has a higher apparent $K_{\rm TC}$, but, for the reasons mentioned earlier, the results obtained at and above 0°C cannot be regarded as valid linear elastic data. It appears that the copolymer can show greater toughness only when it is able to develop an extensive yield zone and that when yielding is suppressed by testing sharply-notched specimens at low temperatures, dispersed elastomeric particles actually weaken the material to a small extent. The angle of the test piece to the flow direction has little effect on toughness and there is satisfactory agreement between the values of ${\rm G}_{\rm IC}$ obtained by the two methods described.

When failure is ductile, the energy U absorbed in tensile impact can be related directly to the ligament area B(W-2a), where B and W are specimen thickness and width and a is crack length. Tests by MP at 23°C on DEN specimens from RP plaques showed that U/B(W-2a) tended to a constant value for 2a/W between 0.65 and 0.85, giving figures of 5.5 kJ/m² for the homopolymer and 8.5 kJ/m² for the copolymer. Similar tests by Hoechst on compression moulded specimens at 23°C, using a 2a/W of 0.33 and an impact velocity of 3m/s, gave values for the homopolymer of 4.6 kJ/m² for 1 mm thick material and 4.1 kJ/m² for 2 mm thick sheet; corresponding figures for the copolymer were 5.1 and 4.6 kJ/m². In 1 mm sheet containing blunt notches of 0.5 mm radius, the energies absorbed per unit ligament area were 15.8 kJ/m² for the homopolymer and 17.4 kJ/m² for the copolymer.

Charpy impact measurements were made on sharply notched compression moulded specimens at 23°C, using Charpy machines equipped with frictionless devices for measuring energy absorbed. All tests were carried out with a span of 40 mm, but width W and thickness B of the bars were varied. Notches were made by means of a sharp milling cutter (BP), which produces a vee-notch, or by pressing a fresh razor blade into the face of the bars (Hoechst.) There was some initial concern about notching procedures as a possible cause of differences in results obtained by the two laboratories. However, further investigation showed that different methods of notching gave similar results for $G_{\rm IC}$ and that the apparent differences arose mainly from the ways in which the raw data were analysed.

The fracture energy G_{TC} was calculated by the method of Plati and Williams (5): the impact energy U was plotted against BWØ, where Ø is a geometrical factor defining the rate of change of compliance. For a linearly elastic material:

$$U - U_{ke} = BW0 G_{TC}$$

where U_{ke} is a kinetic energy term, so that a straight line is obtained, with a slope of G_{IC} and an intercept of U_{ke} . Hoechst also plotted the data in a slightly different way: a plastic zone correction was made by adding 0.10 mm to the measured crack length a before calculating 0, and a constant energy term was subtracted from U so that the resulting line passed through the origin. This procedure had the effect of increasing the linearity between U and BW0, but at the same time, raised the calculated value of G from 1.75 kJ/m² to 2.40 kJ/m² for the homopolymer and from 3.20 kJ/m² to 4.30 kJ/m² for the copolymer. Because of the problems in ensuring that linear elastic fracture has been achieved in these materials, the terms G_{IC} and K_{IC} are used only when instrumented impact tests have been carfied out and linear force-deflection curves obtained. Otherwise, toughness is expressed in terms of the apparent values G_0 and K_0 .

Tests were made on specimens with thickness B of 6, 8 and 10 mm showing no significant effect of thickness on G_0 . It must be borne in mind that the degree of constraint at the tip of the crack is greater in the three point bend (3PB) specimen than in an edge-notched tensile bar, so that plane strain conditions can be expected in 3PB for any given polymer at higher test temperatures than in DEN tension, provided that the specimen thickness is large enough to avoid net section yielding. However, there are indications of non-linearity due to plasticity at the crack tip, especially in the copolymer at 23°C. Results obtained at different specimen widths and thickness, at ageing times ranging from 1 day to 2 year, are given in Figs. 6 and 7 and in Table 2. Apart from the higher G_0 of the copolymer, the most striking effect is that of ageing. Over a period of 2 years in an air conditioned room, the copolymer suffers a 33% decrease in fracture surface energy.

Injection moulded Charpy bars of the copolymer produced in the multi-cavity mould (Fig. 2) were notched to various depths using a fresh razor blade and tested by CIT. Measurements were made at -60° and 23°C on 25 specimens from each of the four positions identified in Fig. 2, and at -25° and 0°C on 12 specimens from each position. Means and standard deviations, calculated as U/BDØ, are given in Table 3. Rather surprisingly, the data show that specimens from position 3, where the notch coincides with the weld, are consistently tougher than those from other positions on the moulding. The fracture resistance falls with temperature, as expected, but at -60°C, G is still well above the value obtained in DEN tension tests (See Table 1).

Specimens measuring 3.7 x 8 x 50 mm were machined from RP double-gated plaques, razor-notched across the narrow face and fractured in 3PB at 23 °C by BW. The homopolymer, which was tested at a loading rate of 5 cm/s, gave a K of 2.5 MPa.m² and showed no significant effect of orientation. The copolymer was tested at a loading rate of 100 cm/s and gave K values of 2.10 MPa.m² for cracks running normal to the flow direction and 1.95 MPa.m² for cracks parallel to the flow direction. The latter figures are consistent with those given in Table 1 for tensile impact tests on the copolymer. The figures for the homopolymer show the influence of increased ductility at lower rates of loading.



Fig. 6 Charpy impact data for homopolymer, obtained by Hoechst. Top: short-term ageing (in days) (X) 1; (o) 2; (+) 4; (\bigoplus) 8; (\square) 16; (\triangle) 130; (\triangle) 135. Bottom: aged for 2 years, with B X W(mm) = (o) 6 X 4; (\bigoplus) 10 X 4; (\triangle) 6 X 6; (\triangle) 10 X 6.



Fig. 7 Charpy impact data for copolymer, obtained by Hoechst. Ageing time (days): (o) 1; (●) 2; (□) 4; (■) 8; (◊) 16; (♥) 130; (△) 135; (X,+) 700.

TABLE 2.	Fracture energy G _Q (kJ/m ²) of polypropylene homopolymer and copolymer determined in impact
	at 23 ^o C. Compression moulded (CM) and injection moulded (IM) specimens.

Type of Moulding	Aging Time	G	Q (kJ/m	2)	W (mm)	Data of
	(days)	at 6	B (mm) 8	= 10		
		HOMOPO	LYMER			
СМ	2	1.57	1.66	1.93	6	BP
СМ	56	1.19	1.39	1.63	6	BP
СМ	1-135	1.75			4	Hoechst
СМ	~ 700	1.35		1.35	4,6	Hoechst
		COPOLY	MER			
СМ	2	4.26	5.08	5.40	6	BP
СМ	1-135	3.20			4	Hoechst
СМ	~ 700	2.15		2.15	4,6	Hoechst
IM Picture Frame		4.17	5.14	5.03	3.8	BP
Picture Fra at weld	me	5.57				BP

(°C)	Position 1	Position 2	Position 3	Position 4
	Mean SD	Mean SD	Mean SD	Mean SD
23	4.27	4.23	5.76	4.26
	0.16	0.31	0.67	0.26
ο	3.26	3.46	4.11	3.55
	0.54	0.32	0.33	0.30
-25	2.66	3.02	3.62	3.37
	0.41	0.55	0.42	0.37
-60	2.60	2.75	2.89	2.41
	0.40	0.46	0.33	0.42

TABLE 3.Fracture energy GQ (kJ/m²) for polypropylene copolymer
determined by Charpy tests on bars from ICI multicavity
mould (Fig. 2), notched after moulding.Fracture is
along weld in Position 3.

TABLE 4.	Mean fracture energy G_0 (kJ/m ²) and 95% confidence limits
	$G_0(max)$ and $G_0(min)$ for the polypropylene homopolymer,
	determined by Charpy impact tests on 6mm wide bars
	machined from RP plaques after 2 years aging. Data of TNO.

Temperature (^o C)	20	0	-20	-50	-70
Mean G _Q	1.17	1.22	1.17	0.81	1.06
G _Q (max)	1.33	1.41	1.40	0.96	1.24
G _Q (min)	1.04	1.05	0.97	0.69	0.91

Charpy specimens machined from RP plaques after 2 years ageing were tested by TNO to determine G_0 of the homopolymer over a range of temperatures. The bars were cut parallel to the flow direction from Type I plaques and across the flow direction from Type II plaques and notched to various depths, 30 specimens, each 6 mm wide, being tested at each temperature. The results are presented in Table 4. They show very little variation with temperature and are all low, confirming that G_0 of polypropylene is reduced by prolonged ageing.

Specimens machined from Type II double-gated RP plaques were used by ICI to determine K_{TC} for the copolymer. Notches of nominal tip radius 10 m were cut into the moulded surfaces of the bars which were tested in three point bending at -70 °C both at low speeds and in impact. The low speed tests were carried out in an Instron tensometer and K_{IC} was calculated from the equation:

$$K_{IC} = \frac{3PS}{2BW^2} Ya^{\frac{1}{2}}$$

where P is the force at fracture, S is span and Y is the geometrical factor. Impact energies were plotted against BWØ to obtain values of $G_{\rm TC}$ and converted to $K_{\rm TC}$ using the equation:

 $K_{\rm IC}^{2} = E G_{\rm IC}^{2} (1-v^{2})$

The tensile modulus was taken as 4.53 GPa at -70 °C and the Poisson's ratio v as 0.4. Checks showed that plane strain conditions were obtained at both test speeds: the plastic zone size calculated from

$$K_{IC}^{2/2\pi \sigma^{2}Y}$$

was small compared with specimen width B and there was excellent linearity between P and BW /SYa². The results are summarised in Table 5: fracture toughness is little affected by either test speed or the

TABLE	5.	Propert	ies of	pol	ypro	py]	lene
copo	lyme	r, measu	ured of	n sp	ecim	ens	cut
from	RP	plaques	at O ^O	and	900	to	the
flow	dir	ection.	Data	l of	ICI	•	

Angle to flow	K _{IC} (MP at - 7	a.m ^{1/2}) 0 ⁰ C	Yield at 0.	Stress 003 s ⁻¹
direction	Slow Ben	d Impact	-70°C	23 ⁰ C
00	2.35	2.27	80.3	52.3
900	2.48	2.63	77.6	54.0

TABLE	6.	Fa	llin	g weig	ht in	npact	t ene	ergies
(J)	at	23 ⁰ C	of	specim	ens f	rom	the	weld
reg	ion	of R	P pl	aques.	Data	of	ICI.	

Plaque	Section of	Impact End	ergy (J)
Type	Weld Tested	Homopolymer	Copolymer
I	Left	2.09	9.90
	Right	2.91	8.32
II	Near Gate Opposite Gate	1.03 3.65	6.87 21.78

angle between the crack and the flow direction. Both K_{TC} and yield stress data indicate a relatively low degree of mechanical anisotropy in the mouldings. As a further check of anisotropy, 150 mm diameter discs were cut from each type of plaque and subjected to three point bending at 23°C with a span of 120 mm. Effective stiffness was calculated, by reference to a RMMA disc (6), at 90° and 0° to the weld line, the ratio of the two stiffnesses being used to define an anisotropy ratio. For the copolymer, the ratio was 0.90 in Type I plaques and 0.82 in Type II plaques. Corresponding figures for the homopolymer were 0.92 and 0.97. As the stiffness of a specimen in flexure is dominated by the properties of the surface layers, which are the regions of highest orientation in an injection moulding, it can be concluded that the cores of the mouldings show even less evidence of anisotropy than these ratios indicate. The ICI figures for K_{TC} at -70°C are somewhat higher than those given in Table 1 for the Same temperature range and the difference is, of course, greater when figures for G_{TC} are compared. Both sets of specimens were machined from RP plaques and there is no obvious reason for the discrepancy.

Charpy impact measurements were made by TNO on blunt-notched specimens over a range of temperatures between -40° and $+40^{\circ}$ C. In one series of tests a semi circular notch of radius 2 mm was machined into 3 x 4 x 50 mm bars cut from compression moulded sheet and the specimens were fractured either 1 or 10 weeks after moulding. In the second series, inserts were placed in the multicavity mould illustrated in Fig. 2, in order to produce injection-moulded bars of the copolymer with moulded-in notches; in these bars, the notch radius was 0.25 mm. The results are presented in Figs. 8 and 9. Figure 8 shows that the



Fig. 8 Charpy impact energy of compression moulded polypropylene bars with 2mm radius notch. Ageing time: (o, △) 1 week; (•, ▲) 10 weeks. Data of TNO.



Fig. 9 Charpy impact energy of copolymer specimens with moulded-in notch from ICI multicavity mould. Data of TNO. Positions as shown in Fig. 2: (o) 1; (Δ) 2; (o) 3; (\blacktriangle) 4.

copolymer is tougher than the homopolymer at all test temperatures, in contrast to the results of fracture mechanics tests. Changes in density over the 9 week interval between tests do not significantly affect the impact strength of the compression mouldings. Because of their sharper notches, the injection moulded bars have lower impact strengths than the compression mouldings. There is no systematic difference in impact strength between bars from the four different positions in the mould and it must therefore be concluded that neither orientation nor the presence of a weld line has any significant effect upon notched Charpy impact energy. This further confirms the conclusion of the first report in this series (1).

Dart impact tests on plaques

Instrumented falling weight tests were performed by ICI on rectangular specimens machined from the RP double gated plaques. Specimens were freely supported on a 40 mm diameter steel ring and struck at 3 m/s with a 12.5 mm diameter steel ball. All plaques were struck on the face marked with the ejector pins, so that these defects were in compression. Type I plaques of copolymer were tested at 0°C and Type I and II plaques of homopolymer were tested at 23°C. The results are summarised in Fig. 10. Both in homopolymer and copolymer, the weld region gives lower impact energies than the remainder of the plaque in Type I mouldings, where melt fronts meet head on. The weld region is also weaker than the rest of the plaque in Type II mouldings, where the flow is parallel, but the homopolymer also fractures at low impact energies at some other points in the moulding and the weld does not stand out so clearly as it does in Type I mouldings. Differences in the location of ejector pin marks could account for these observations.

Because of differences in test temperature, it is not possible to compare the two polymers directly on the basis of these measurements. In a subsequent experiment, ICI studied the weld region of both homopolymer and copolymer at 23°C, using the same test procedure. In the case of Type I plaques, the right and left hand sections of the plaque are compared, whilst in Type II plaques the upper half of the



moulding, nearer the gates, is compared with the lower half of the moulding. Ten specimens were tested from each location. The results are shown in Table 6. The copolymer is obviously much tougher on average than the homopolymer in all four locations, although some low individual values of impact energy are recorded for both halves of the Type I plaque and for the area of the Type II plaque nearest to the gates. The area of the Type II plaques distant from the gates is substantially tougher than the area nearer the gates and in the case of the copolymer gives 100% high energy failures.

Monsanto studied the impact behaviour of the RP plaques using an instrumented driven-dart machine, in which a 38 mm diameter hemispherical striker is driven through the specimen at a constant speed of 2.64 m/s. Each plaque was cut into three sections, labelled left, centre and right as viewed from the ejector pin side of the moulding. The cutting plan is shown in Fig. 11. Fifteen specimens were taken from each position, clamped between rings of inside diameter 57 mm and tested at 22°C. The results are summarised in Fig. 12a and 12b.

The single consistent feature is that the copolymer is tougher than the homopolymer. In some of the tests on the copolymer, none of the specimens showed any sign of a break. In general, the lowest energy failures occur at the weld line but this is not always the case: when the impact is on the ejector pin side of the moulding, the weld region of Type I plaques of the homopolymer is substantially tougher than the remainder of the moulding. The fracture of Type II plaques of the homopolymer is 100% brittle when the impact is on the smooth side of the moulding (opposite the ejector pin side) and the corresponding series of tests on the copolymer also gives 100% low energy failures in the centre and left hand sections of the plaque. Other conditions show either a mixture of high and low energy failures or, in the case of the copolymer, 100% high energy failures.

The ejector pin marks on Type II plaques are at, or close to, the point of impact in this test. Furthermore, the marks are prominent and can certainly be considered to constitute defects from which cracks can nucleate. The observation that impact energies are low when Type II plaques are struck on the opposite face, so that the ejector pin marks are in tension, can be explained in this way. Much higher energies are needed to fracture the specimens when they are struck on the face marked by the ejector pins, especially in the case of the copolymer.



Fig. 11 Cutting plan for Monsanto driven dart specimens.



Fig. 12 Driven dart impact data for specimens from Rhone-Poulenc plaques. Note difference in energy scale between homopolymer and copolymer. Specimens are struck on upper face. L, C and R as defined in Fig. 11. Data of Monsanto.

CONCLUSIONS

The following conclusions can be drawn from these results:

(a) Problems remain in determining $G_{\rm LC}$ and $K_{\rm LC}$ of polypropylene homopolymer and copolymer under impact conditions with any degree of precision. Both within laboratories and between laboratories, there are differences of up to 30% in the measured values of $G_{\rm LC}$, in tests on identical mouldings fractured at the same test temperature and at the same time after moulding. On the basis of a limited study, it appears that these differences cannot be attributed to differences in notching procedure, provided that adequate care is taken. The application of a plastic zone correction can substantially alter the value obtained for $G_{\rm LC}$. However, the major source of variation is probably the scatter inherent in the impact energy readings recorded in individual tests on sharply notched specimens. In order to ensure that force-deflection curves are linear, and valid $G_{\rm IC}$ or $K_{\rm IC}$ data are obtained, instrumentation is essential.

(b) Both in the homopolymer and in the copolymer, the impact fracture energy G at 23°C decreases with time as a result of physical ageing which is also reflected in an increase in density with log (time). These changes continue to affect properties over periods greater than 4 months.

(c) At 23°C, the fracture resistance of the copolymer, as measured by G and K_O, is substantially higher than that of the homopolymer. However, the reverse is true at -40° and below, where the extent of yielding at the crack tips is much reduced. A similar conclusion was reached by Fernando and Williams (4).

(d) Charpy tests on sharply-notched specimens show no evidence of weakness within the weld region, nor of significant anisotropy with the RP plaque mouldings. Flexural stiffness tests support the view that the mouldings are substantially isotropic. It is, therefore, concluded that the large variations in fracture behaviour observed in dart impact tests on mouldings are due to the presence of surface defects rather than internal weaknesses in the materials.

(e) The weld constitutes an important defect in Type I mouldings (head on flow) and near the gate in Type II mouldings (parallel flow). However, other defects, including ejector pin scars, can also substantially reduce the impact resistance of the moulding.

(f) The toughness of the polymer and the size of defects present in the moulding both have an important influence upon the impact energy of an unnotched moulded item.

REFERENCES

- 1. S. Turner, Pure and Appl. Chem. 52, 2739 (1980).
- 2. T. Casiraghi, Poly. Eng. Sci., 18, 833 (1978).
- 3. ASIM SIP 410.
- 4. P. L. Fernando and J. G. Williams, <u>Poly. Eng. Sci.</u>, <u>20</u>, 215 (1980).
- 5. E. Plati and J. G. Williams, Poly. Eng. Sci., 15, 470 (1975).
- R. C. Stephenson, S. Turner and M. Whale, <u>Plast. Rubb.</u>, <u>Mat.</u> <u>Appl.</u>, <u>5</u>, 7 (1980).