IMPACT PROPERTIES ANALYSIS OF ROTATIONALLY MOLDED POLYETHYLENE AND POLYPROPYLENE FOR A WIDE RANGE OF TEMPERATURES

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Abstract

Rotational molding is an established and growing manufacturing method for large, hollow plastic components. In this work the impact properties of rotationally molded Polyethylene (PE) and Polypropylene (PP) were tested at temperature in the range of -40 ºC to 30 ºC. Dynamic mechanical thermal analysis (DMTA) was performed to analyse the measured impact properties of PP and PE plastics. For PP, a very good relationship was found between peak impact strength and the loss modulus curve obtained in DMTA analysis. A relationship ~~among~~ between density, β peak height and peak impact strength was found for PE which is different from previous findings in the literature. It is concluded that further work should focus on developing an understanding of the PE material's microstructure in order to more fully understand its impact properties.

Introduction

Rotational molding is one of the fastest growing processes in the molding of plastics due to its simplicity, stress free parts production and relatively uniform thickness distribution. This makes it particularly suitable for large, hollow plastic products [1-3]. In the rotational molding industry, generally impact properties are measured to check the quality of the products for using in different applications. Temperature has a direct effect on fracture behavior in impact loading and can change the fracture mode [4]. Therefore researchers have attempted to find the relationship between thermal transitions of roto-molded plastics and the impact properties [5, 6].

Dynamic mechanical thermal analysis (DMTA) is generally used to characterize the thermal transitions of the materials that are created by chain movements in the materials. It can identify the storage (E') and loss modulus (E") for the elastic and viscous responses of a viscoelastic materials respectively. tanδ is the ratio of the loss modulus to storage modulus.

There are three transitions for semi-crystalline polymers particularly in PP and PE. α, β, γ transition peaks normally represent chain motion in the crystalline portion, glass transition and amorphous region respectively [7]. Different transitions of rotationally molded PP were investigated before and impact behavior was described based on these transitions [4]. PP is a brittle material because of its high glass transition or β transition temperature. To reduce this brittleness co-polymerization was carried out with ethylene to lower the β transition temperature [8]. The β transition has a correlation with high impact strength of PE and was found in the region between the high impact strengths obtained at low temperatures and the lower impact strengths obtained at high temperatures [6]. A numerical relation was also developed ~~among~~ between peak impact strength, tanδ and the β transition region of polyethylene [5]. The density of the materials is directly related to the β peak height. Previous work has generally shown higher β peak height (loss modulus) results in better impact resistance for PE, however a recent paper by Pick et al. [6] has shown a correlation between lower β peak height and increased impact strength for higher density rotationally molded PE and this warrants further investigation.

In this work, the drop weight impact properties of rotationally molded PE and PP were measured at 10 ºC intervals from -40 to 30 ºC. DMTA was carried out to correlate the thermal transition with impact properties of tested PP and PE. Density, loss modulus, β peak height were checked for both materials and described particularly for PE to relate with the measured impact properties.

Experimental Methods

Materials

Rotationally mold grade PE and PP were used in this study, supplied by the Matrix Polymers Ltd. Material details are given in Table 1. PE and PP materials are identified by a code starting with PE and PP respectively, followed by a number (I, II) e.g. PE-I = Polyethylene-I.

Table 1 Material details[1].

No.	Code	MFI g/10 min	Density g/cm^3	Yield Stress MPa
1.	PE-I	3.50	0.939	17.7
2.	PE-II	3.50	0.949	21.5
3.	PP-I	25.00	0.902	25.5
4.	PP-II	30.00	0.902	23.5

[1] Materials and details are supplied by Matrix Polymers, UK. MFI = Melt Flow Index.

Rotational Molding

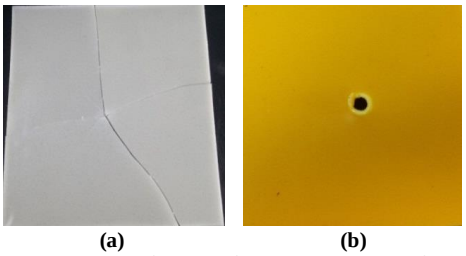
The impact test samples were made using a Ferry Roto-speed Carousel type rotational molding machine at Matrix Polymers Ltd. UK facilities. Moldings were produced in a 300 mm steel cube mold. A shot weight of 1.5 kg was used in each trial to produce moldings with a nominal wall thickness of 3 mm. All the moldings were produced under the following conditions- moldings were heated up in an oven at 250 ℃ for 15 minutes and then the mold was removed from the oven and cooled with fans for 15 minutes and finally de-molding was occurred.

Impact Testing

Impact test were carried out with an instrumented falling weight impact testing machine according to ASTM-D 3763 – 02 standards. Impact samples were machined from molded plastics into 125×125 mm squares and placed on the sample holder with a circular window cut-out of 90 mm diameter in the center of the holder. The impactor which was used to strike the clamped specimens, is a hemispherical indenter with a 12 mm diameter. A piezoelectric impact force sensor of maximum loading capacity of 22.4 kN is used to measure impact force over time for each test. The total falling mass of the impactor for these tests is 9.1 kg (including impactor and crosshead mass).

A high resolution oscilloscope (Picoscope IEPE 4242) was used to acquire the data generated in the impact event. A force-time graph was drawn for each of the test. Peak impact strength was calculated from the area under the curve up-to highest point in the impact curve while total strength was found from the area under the whole curve. The samples were impacted from a height of 1 m with an approximate 4.4 m/s impact speed. Five impact samples for each material were tested 10 ºC intervals from -40 to 30 ºC.

Samples were conditioned in an environmental chamber (Votsch, VCL 4003) at each temperature for 3 hrs before testing.



(a) **(b)**

Figure 1 (a) Brittle fracture of PP-I and (b) ductile fracture of PE-I from the drop weight impact test.

Dynamic Mechanical Thermal Analysis (DMTA)

A METLER TOLEDO DMTA machine was used to conduct the dynamic mechanical thermal analysis of each material. Samples of 35×10×3 mm were placed in the dual cantilever mode in the DMTA machine. Samples were tested at 0.005 strain from -150 to 100 ºC. The heating rate and frequency were 2 ºC/min and 1 Hz respectively.

Results and Discussion

Polypropylene (PP)

The average peak impact strength of PP materials is mentioned in Table 2. The failure mode over all the temperatures is observed to be brittle (see Figure 1). From Figure 2, it is seen that the impact strength is constant up-to 10 ºC for both PP materials tested. Above 10 ºC, the impact strength is found to increase rapidly for PP-II compared to PP-I.

Table 2 Average peak impact energies of PP-I and PP-II at the range of temperatures.

Temp-erature (ºC)	PP-I		PP-II	
	Energy (J/mm)	Standard deviation (±)	Energy (J/mm)	Standard deviation (±)
-40	0.010	0.008	0.015	0.007
-30	0.012	0.005	0.015	0.005
-20	0.010	0.004	0.014	0.004
-10	0.025	0.015	0.034	0.009
0	0.036	0.020	0.037	0.015
10	0.036	0.016	0.036	0.020
20	0.095	0.024	0.281	0.092
30	0.070	0.010	0.600	0.135

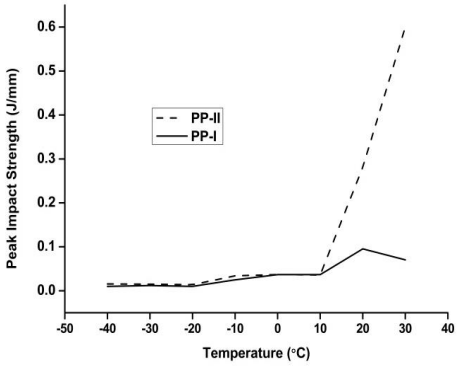


Figure 2 Peak impact strength of PP-I and PP-II.

In the loss modulus graph (Figure 3), major transitions are found at 6ºC and -23 ºC for PP-I and PP-II materials respectively. For PP-I some other peaks are also found at lower temperatures that could be related to its structural arrangement. For PP, the relationship between loss modulus and peak impact strength is very evident which was also observed in a previous work [9]. Brittle fracture with lower peak impact strength was found at temperatures lower than the β-transition for both of the PP materials. After the β transition the peak impact strength increases which is prominent for PP-II although no change in fracture mode was found.

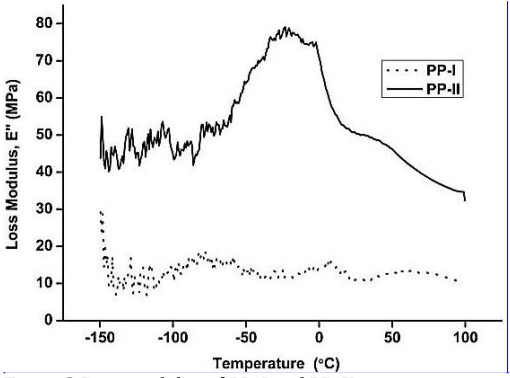


Figure 3 Loss modulus of PP-I and PP-II.

Polyethylene (PE)

Peak impact energies are shown in Table 3 for PE-I and PE-II. The mode of fracture of both PE materials was ductile at all temperatures. One example is given in Figure 1. From Figure 4, it is found that PE-II has better impact properties than PE-I. It also can be seen that the peak impact strength of both PE samples reduces with temperature from -40 to 30 ºC. PE-II varies less, only 0.84 J/mm between -40 to 30 ºC whereas PE-I shows more than 1 J/mm reduction in the same temperature range.

Table 3 Average peak impact energies of PE-I and PE-II at a range of temperatures.

Temp-erature (ºC)	PE-I Energy (J/mm)	PE-I Standard deviation (±)	PE-II Energy (J/mm)	PE-II Standard deviation (±)
-40	1.953	0.174	2.150	0.180
-30	1.500	0.200	2.155	0.237
-20	1.400	0.167	2.350	0.240
-10	1.143	0.135	1.940	0.165
0	1.473	0.140	1.443	0.156
10	0.903	0.123	1.670	0.115
20	0.735	0.100	1.190	0.105
30	0.750	0.173	1.310	0.110

The MFI values of PE-I and PE-II are same (see Table 1), though these materials are different in density. PE-II has higher density and shows better impact properties. Normally density increases with crystallinity and reduces impact strength, however this is not observed in this work.

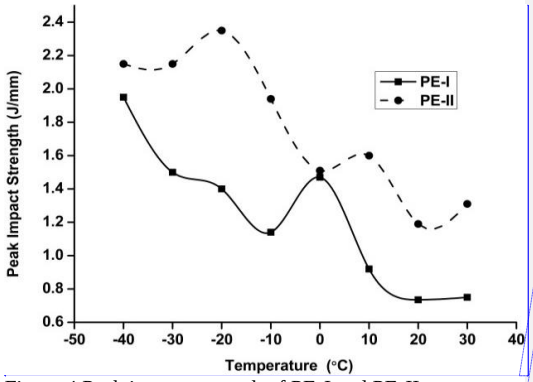


Figure 4 Peak impact strength of PE-I and PE-II.

In Figure 5 the loss modulus of dynamic mechanical analysis of PE-I and PE-II is presented. Three different peaks are clearly seen at three different temperatures. PE-II shows α peak at a higher temperature compared to PE-I with higher intensity. The high intensity of the α-relaxation peak increases with crystallinity or crystal thickness [7] which also supports its high density compared to PE-I. PE-I and PE-II show β relaxation peaks at -48 ºC and -41 ºC respectively. Lower density of PE-I describes the β relaxation peak at lower temperature.

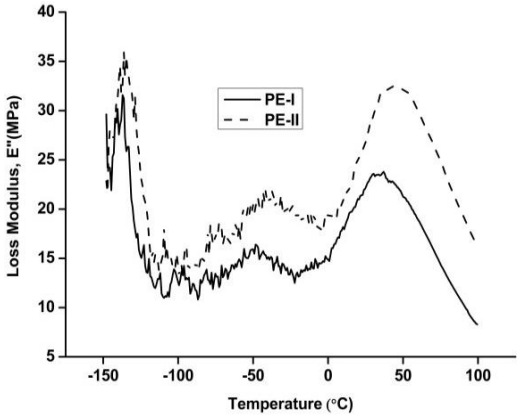


Figure 6 Loss modulus of PE-I and PE-II.

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Pick et al. [6] found a lower β relaxation peak height for higher density PE that showed higher peak impact properties. In this work, higher density of PE increases β relaxation peak height which results in higher peak impact strength. This observation is different to that of Pick et al [6] . However, it is not clear which factor causes higher or lower β relaxation peak height that is correlated to high impact strength. This could be due to the crystal structure and microstructure arrangements in the materials. To find out the important factors related to β relaxation peak height detailed further investigations into ~~on~~ the microstructurale~~-~~ arrangements is necessary.

Conclusions

It is seen that polyethylene with higher density shows a higher β relaxation peak height in the loss modulus curve. A higher β peak height results in better impact properties. However, the ~~finding~~ relationship betweenabout density, β peak height and impact strength in this work is different than that of previously reported by Pick et al. [6]. Some other factors could be related to β peak height than density and this need to be investigated in the future. Polypropylene shows the expected results for loss modulus and impact strength properties.

Acknowledgement

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