NANO FIBRILLATED HIGH MODULUS DUCTILE (HMD) TECHNOLOGY IN ENVIRONMENTALLY SUSTAINABLE XENOY IQ* RESINS

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Abstract

Recently, General Electric Plastics launched a series of High Modulus Ductile (HMD) products, as an expansion to the Xenoy product line. In these HMD products, a highly fibrillated nano network is combined with state of the art mineral filler technology, allowing for retention of impact and tensile properties whilst increasing the modulus of molded articles. We have been successfully able to incorporate this technology in the Xenoy* (PC/PBT and PC/PET) resin, which has resulted in superior chemical resistance, low CTE, excellent tensile strength, fatigue, and low temperature ductility.

We will present a case study where HMD technology was combined with our environmentally sustainable, low carbon footprint Xenoy iQ* resin offering excellent part performance, lighter weight, and increased first pass yield during processing.

Introduction

PC/Polyester alloy is a very important class of thermoplastics due to its excellent balance of impact performance and chemical resistance. It has been widely used in automotive, power tool, and OVAD (Outdoor Vehicles And Devices) applications. Typically, PC/PBT blends have a modulus about 2 Gpa and a notched Izod impact strength of >500 J/m. In the quest for lighter weight leading to higher fuel efficiency, it is becoming more important to attain a higher modulus with similar impact performance as a traditional PC/PBT blends. Traditional way of getting the extra modulus is through the addition of mineral filler. However, this usually leads to the sacrifice of impact performance. For example, a PC/PBT blend with 15 percent mineral filler has a tensile modulus of 3.5 Gpa and a Notched impact strength of ~80 J/m. On the other hand, an increase in the level of impact modifier increases impact at the expense of modulus. The new GE Plastics proprietary HMD technology changes this paradigm, where a combination of higher modulus and impact can be gained at the same time as conceptually demonstrated in Figure 1. The high stiffness was achieved through the use of mineral filler. Nano fiber networks were introduced to the material for added impact performance.

PC/PBT blend with HMD technology provides superior modulus/ductility balance, high heat, excellent fatigue resistance, low creep, low CTE, good appearance, and excellent chemical resistance. The superior modulus/ductility balance enables weight reduction via thinner walls. It also provides robust ductile products, dimensional heat stability, extended durability and excellent fit & finish over wide temp range. This material can also be painted or use as molded-in-color. It is a good fit for portable power tools, automotive body panels, door handle, spoiler, safety shoe caps and energy absorbers. In addition, the HMD technology can be combined with the Xenoy iQ* resin (made by alloying PC with PBT resin that is manufactured via depolymerization of post-consumer PET followed by polymerization into PBT) thereby providing customers with performance and environmental sustainability and low carbon footprint.

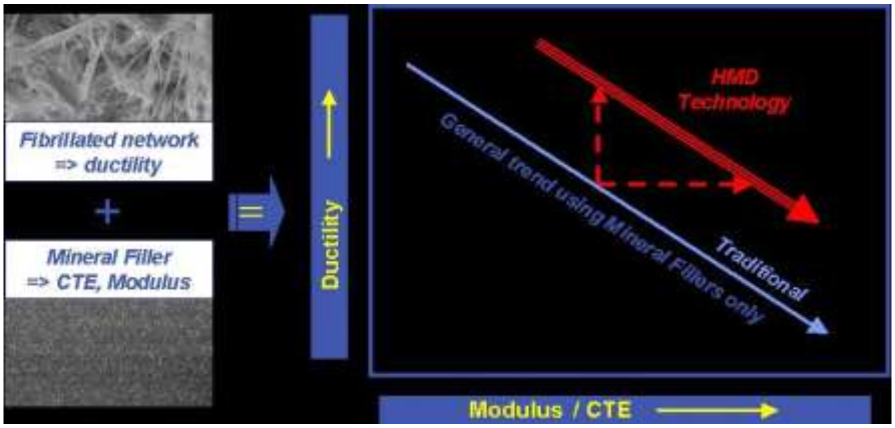

Figure 1. HMD technology concept demonstration

This study exhibits the use of environmentally responsible PC/PBT blend in an automotive door handle application.

Experimental

Material development was done on a 40mm Werner Pfleiderer twin-screw extruder. The extrusion line was of a design similar to that found in larger compounding plants, although the smaller barrel diameter and resultant feed rate capacity make the line suitable for experimentation at the pilot scale. The feeding of all materials occurred at the feed throat of the extruder and was accomplished via the use of loss-in-weight auger or belt feeders attached to the line. Typically, each feeder carried only one raw material. The exceptions are materials present in small percentages. These materials are combined together as a blend, prior to extrusion, and fed as one combined material on a single belt feeder. No unusual compounding conditions were employed during the production of these materials. The combined feed rate of all feeders was 300 lbs per hour and the screw speed was 480 RPM. The extrudate was cooled under water and cut into pellets. The pellets were typically dried for 3-4 hours at 110°C in a forced air-circulating oven prior to injection molding. Test parts were injection molded on a van Dorn molding machine with a set temperature of approximately 250°C.

Tensile properties were tested on Type I tensile bars at room temperature with a crosshead speed of 50 mm/min according to ASTM D638. Notched Izod testing was done on 3 x 1/2 x 1/8 inch bars according to ASTM D256 at multiple temperatures with pendulum energy of 5 lbf/ft. The flexural bars were tested at a speed of 1.27 mm/min as per ASTM D790. HDT was tested at both 0.455 and 1.82 Mpa per ASTM D 648. Multiaxial impact testing sometimes referred to as instrumented impact testing, was done per ASTM D376 using 4 x 1/8 inch molded discs. The total energy absorbed by the sample is reported in Joules. Shrinkage was done according to Global GE Shrink method on 4x0.125 in discs.

Tensile creep test were performed according to ISO 899 on ISO MPTS tensile specimens. Specimens were tested at least one month after molding and at most four months after molding. Samples were tested at room temperature or an elevated temperature under different stress. At 23 ºC (RT) the humidity is controlled at 50 percent RH. When specimens were tested at elevated temperature they were held at this temperature for 24 hrs, prior to applying the loads. Strain was recorded as a function of time.

Tensile fatigue test was carried out using standard ASTM Type 1 tensile bars. The test bar is clamped in a (servo-hydraulic) universal testing machine and it is subjected to a cyclically varying load at a specified frequency. The number of cycles to failure at various mean stress levels is plotted to get the fatigue curve. This test was done in an environment of 23 ±2^{o}C, 50 ±5 percent RH and specimens were conditioned in the testing environment for 48 hrs. The test is load-controlled, the load being varied in a sinusoidal waveform between 100 percent and 10 percent of the nominal stress level at a frequency of 5 Hz. Normally two bars per stress level were tested and the individual values were taken. Data from specimens that fail prematurely may be discarded. Specimen rupture was deemed as failure.

Paint adhesion was evaluated under two different methods: Steam jet and crosshatch. Parts were painted with Wörwag water based Base coat Black Jack, (10-15 microns) with a drying for 15 min @80^{o}C and Wörwag Solvent based Clear coat, (25-30 microns) with a curing for 45 min @ 80-85^{o}C. The steam jet test was used to determine resistance of paint layers to the influences of steam with a specific temperature, pressure and with a prescribed nozzle. The adhesion loss was specified in 0= Ok, No loss of adhesion, and 5 =large area of adhesion failure. Conditions used in this study are listed in Table I. In the cross-hatch test, a lattice pattern is obtained by first making six parallel cuts with the cutting tool. The lattice pattern is then covered with tape (Tesa 4651). The tape is removed quickly using a little extra force. The adhesion was visually assessed according to the standards in Figure 2.

Table I. Conditions for Steam jet tests

	HDW A	HDW B
Pressure (Bar)	65 +/- 1	65 +/- 1
Distance Nozzle Tip / painted surface	10 cm	15 cm
Duration (sec.	60	60
Temperature (C)	60 +/- 1	60 +/- 1
Nozzle Type	std	"DCX"

HDW B is acc. SMART spec. 96002

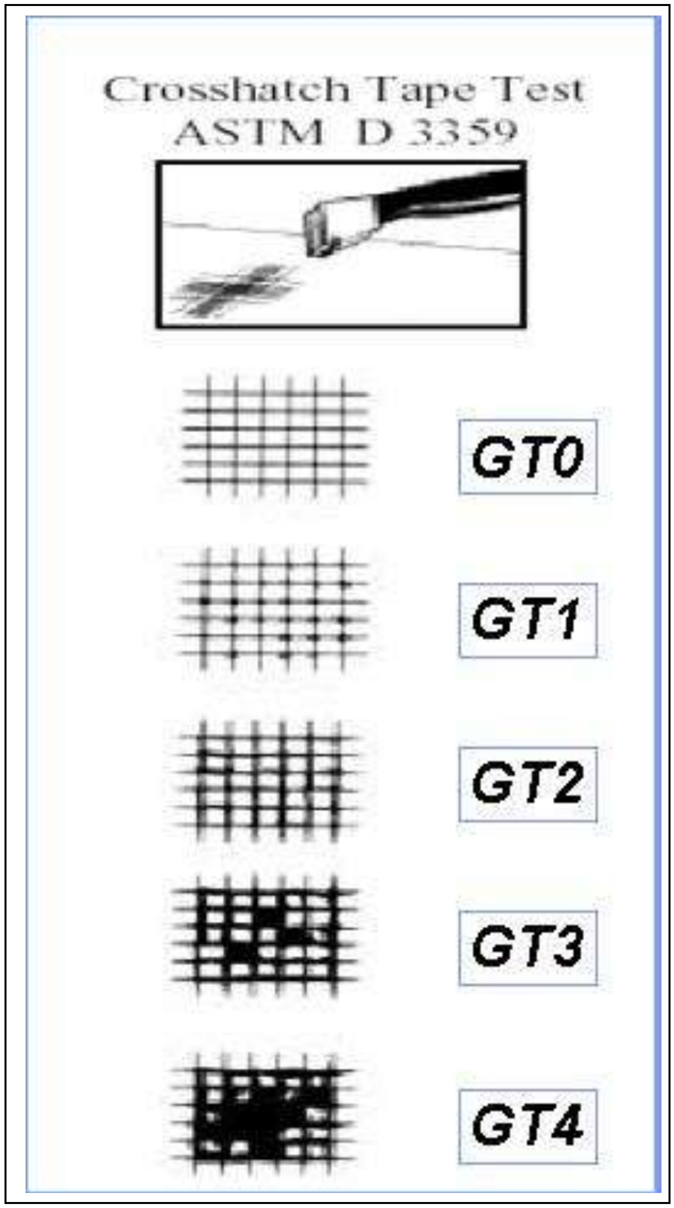

Figure 2. Crosshatch test visual evaluation standard

Results & Discussion

The property data of this material is listed in Table II. These data suggest very good modulus and impact balance. With a tensile modulus as high as 4 Gpa, a notched Izod impact of 250 J/m could be obtained using the pilot scale extruder described in the experimental session. This represents more than twice of the impact strength for normal Xenoy with slightly less modulus. Due to the mineral content, this formulation also has high HDT under high pressure.

Paint adhesion test - Paint adhesion tests for typical HMD PC/PBT formulations are shown in Table III. Good performance was observed for both the steam jet test and the crosshatch test.

Superior creep performance - Figure 3 compares the creep performance of HMD PC/PBT blend (EXXY0137) vs. a typical mineral filled Xenoy resin. Deformation of EXXY0137 was minimal at room temperature at 10 Mpa and 25 Mpa, similar to the other PC/PBT grade. However, at elevated temperature, the HMD PC/PBT (EXXY0137) showed much less creep than the comparison grade at 25MPa.

Table II. Preliminary Physical property datasheet

MECHANICAL	Unit	Value
Tensile Stress, yld, Type I, 5 mm/min	MPa	62
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Tensile Strain, yld, Type I, 5 mm/min	%	3.9
Tensile Strain, brk, Type I, 5 mm/min	%	130
Tensile Modulus, 5 mm/min	MPa	4000
Flexural Stress, yld, 1.3 mm/min, 50 mm span	MPa	104
Flexural Modulus, 1.3 mm/min, 50 mm span	MPa	3600
Izod Impact, notched, 23°C	J/m	250
Multiaxial impact, total energy, 23°C	J	63
Multiaxial impact, total energy, -20°C	J	64
Multiaxial impact, total energy, 23°C	J	59
Vicat Softening Temp, Rate B/50	°C	127
HDT, 1.82 MPa, 3.2mm, unannealed	°C	100
Specific Gravity	-	1.29
Mold Shrinkage, flow, 3.2 mm	%	0.7 - 0.9
Melt Volume Rate, MVR at 265°C/5.0 kg	cm^3/10 min	9

Table III. Paint adhesion test

<table>
<tr><th rowspan="4">Melt Temp for molding (C)</th><th colspan="6">Steamjet test</th><th rowspan="4">Cross Hatch</th></tr>
<tr><th colspan="4">HDW A</th><th colspan="2">HDW B</th></tr>
<tr><th colspan="2">Part 1</th><th colspan="2">Part 2</th><th rowspan="2">Part 1</th><th rowspan="2">Part 2</th></tr>
<tr><th>1</th><th>2</th><th>1</th><th>2</th></tr>
<tr><td>265</td><td>OK</td><td>OK</td><td>OK</td><td>OK</td><td>OK</td><td>OK</td><td>OK</td></tr>
<tr><td>275</td><td>OK</td><td>OK</td><td>OK</td><td>OK</td><td>OK</td><td>OK</td><td>OK</td></tr>
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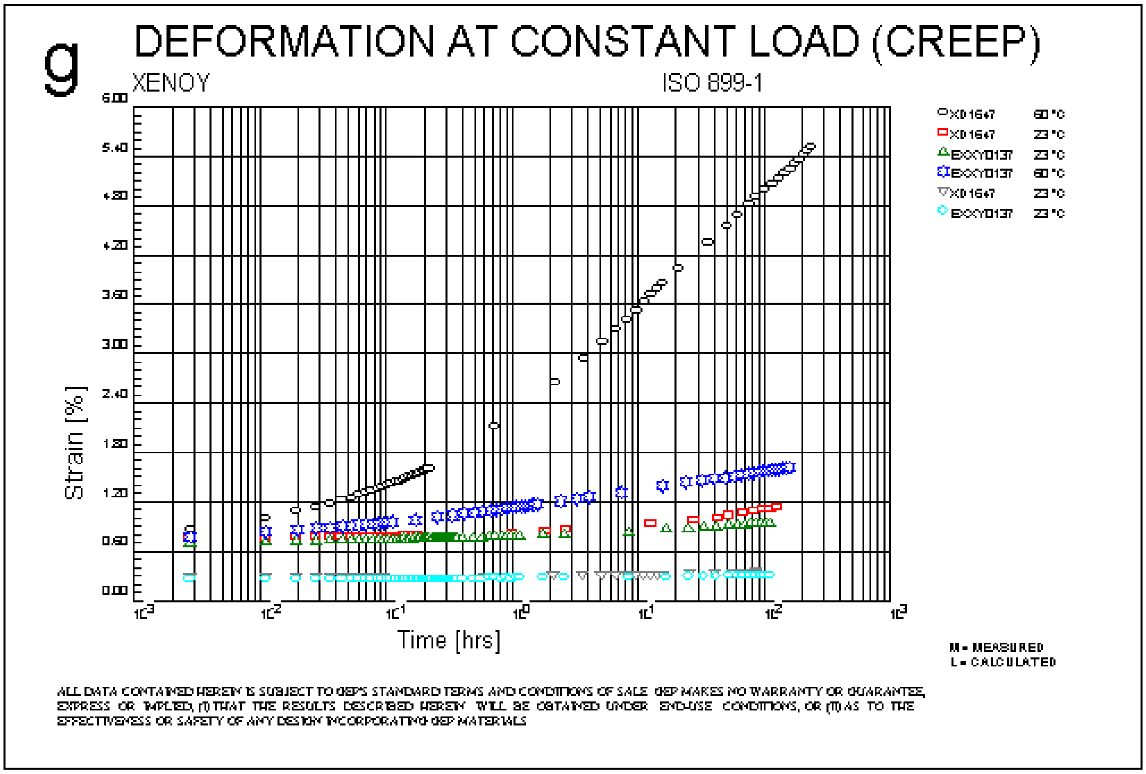

Figure 3. Creep Performance

Fatigue tests (Figure 4) – The PC/PBT blend EXXY0137 has a superior performance than a typical mineral filled PC+PBT resin as shown in Figure 4. At 30 bar, EXXY0137 shows 3X-5X the number of failures as compared to a typical mineral filled PC&PBT resin.

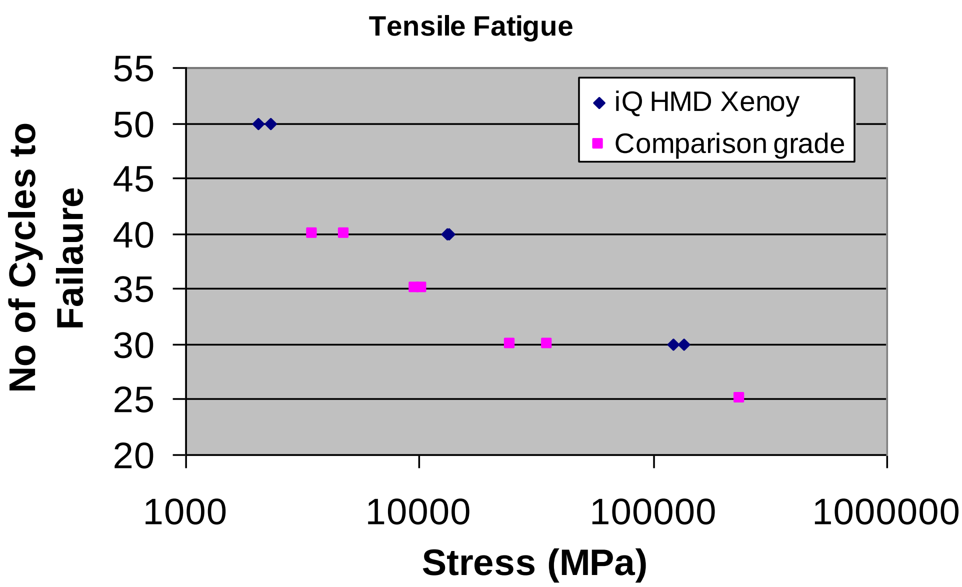

Figure 4. Fatigue Performance

Conclusions

Typical PC/Polyester alloys have certain modulus and impact balance. HMD technology pushed the envelope and can provide higher modulus at the same impact level or higher impact performance at the same modulus level. It also shows better fatigue and creep properties than normal PC/polyester blends. The heat performance is also better with HMD technology than similar blends not using this technology. It also demonstrated excellent paint adhesion properties. Although not elucidated in this paper, the HMD technology has also been used in molded-in-color applications.

Acknowledgements

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