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Linking knowledge with action

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Report on the Oxidative Stability of Polypropylene Base Material

A small sample of a black fabricated product molded from a material identified as a polypropylene was provided for an evaluation of projected durability at application temperatures expected to reach a maximum of 70-80^{o}C. Because the application location does not involve exposure to sunlight, the primary mechanism of concern that would contribute to reduced lifetime is oxidation. Therefore, the oxidative stability of the material was characterized using oxidation induction time (OIT) tests. Results obtained at elevated temperatures were then extrapolated using an Arrhenius relationship that provides a prediction of performance at the anticipated maximum operating temperatures.

In order to provide sound extrapolations, it is important that all the temperatures used to characterize the oxidative stability of the material maintain the material in the solid state. Therefore, the first step in the experiment involves establishing the crystalline melting point of the material. This is accomplished by differential scanning calorimetry (DSC). A sample taken from the part was heated in nitrogen from room temperature to 250^{o}C at a rate of 10^{o}C/minute, cooled at the same rate back to 25^{o}C, and then reheated to 275^{o}C. The results are provided in Figures 1 and 2 as two plots of heat flow versus temperature for first heat, cooldown, and second heat. The sample shows a major melting point at 164^{o}C that is characteristic of polypropylene. A smaller melting point at 124^{o}C indicates the presence of a small amount of polyethylene.

Given a melting point of 164^{o}C, oxidation induction time tests were conducted by differential scanning calorimetry (DSC) at three temperatures; 160^{o}C, 155^{o}C, and 150^{o}C. Samples were heated in nitrogen to the target temperature at a rate of 20^{o}C/minute and held isothermally at the test temperature for five minutes. The atmosphere was then switched to oxygen to promote consumption of the antioxidant package in the material. The oxidation induction time (OIT) is measured as the interval between the switchover time and the onset of the exotherm indicating the initiation of oxidative degradation. These results are plotted as heat flow versus time and provided in Figures 3 through 5. Because the required time to heat and equilibrate all the samples is 16 minutes, the OIT represents the total time to oxidation minus this 16 minute conditioning time.

The results show that as the test temperature declines the OIT increases. Table 1 summarizes the actual OIT values and the corrected values associated with the model used to fit the data from the three experiments. Figure 6 provides a semi-log plot of OIT as a function of temperature. The tests show that the OIT increases by a factor of nearly 2.5 for a decline in temperature of 10^{o}C. This represents an exponential relationship so that each subsequent decline of 10^{o}C increases the OIT by a factor of 2.5. Table 2 shows the projected lifetimes for the material at these lower temperatures and Figure 7 presents the accompanying plot.

Table 1 – OIT Results
(All Results in Minutes)

Temperature (^{o}C)	OIT - Actual	OIT - Model
160	1365	1250
155	1850	2050
150	3406	3200

Table 2 – Extrapolated OIT Results

Temperature (^{o}C)	Extrapolated Lifetime In Pure Oxygen (minutes)	Extrapolated Lifetime In Pure Oxygen (hours)
160	1250	21
150	3200	53
140	8000	133
130	20000	333
120	50000	833
110	125000	2083
100	375000	6250
90	937500	15625
80	2343750	39063
70	5859375	97656

Tests were run in pure oxygen. Atmospheric conditions involve an oxygen concentration of 21% and work performed previously shows that the reduction in oxygen concentration from 100% to 21% extends the oxidation induction time by a factor of 3.6. This adjustment produces a predicted lifetime of 140,625 hours or 16.28 years at 80^{o}C and 351,562 hours or 40.69 years at 70^{o}C.

It should be noted that extrapolations performed across seven to eight decades of temperature have the potential to introduce significant deviations from predictions. These errors can result in actual lifetimes that are shorter or longer than predicted. A conservative estimate has been applied here by reducing the acceleration factor per decade of temperature from 2.5 to 2.3. This is a well established multiplier used in the medical device industry and it reduces the predicted lifetime by 50% to 8.14 years at 80^{o}C and 20.35 years at 70^{o}C. However, this material produces an extremely high actual OIT result at 160^{o}C compared to other materials with known capabilities to perform at elevated temperatures for extended period of time. Therefore, the longer projections are within reasonable limits. The incorporation of the polyethylene into the material improves the oxidative stability of the compound.

Polypropylenes that are exposed to elevated temperatures can undergo other structural changes that cause a change in properties. The most important of these is residual crystallization. This will increase the strength and stiffness of the material while reducing its toughness. This mechanism has not been modeled here. However, the largest reduction in properties for polypropylenes arises from oxidation. Therefore, by modeling this mechanism, the largest contributor to performance reductions has been accounted for.

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8/5/2011