Technical Notebook

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PolyDyne micronized rubber powder (MRP) of 80, 140 and 200 mesh particle sizes were evaluated in a model compound consisting of 40 PHR Natural Rubber and 60 PHR high cis polybutadiene rubber. The model compound contained 60 PHR N550 carbon black and 21 PHR oil.

The MRP was compounded at 5 and 10 percent over batch weight. One series of the 140 mesh evaluation included recommended counter measures of increased sulfur and reduced accelerator while all other compounds in this study were not adjusted from control levels. Table 1 illustrates the recipes used.

Compounds were mixed on a 1.6 liter variable speed internal mixer using a 3 pass mix. The MRP was added with the carbon black in the first pass. Tables 2-4 show the mix procedure for each of the mixing passes used. Compound samples were cured to a 100 percent state of cure at 160°C and subsequently evaluated using the following guide test procedures:

1. MDR2000 Rheometer—ASTM D 5289 @ 160°C
2. Tensile, Elongation, Modulus—ASTM D 412, unaged and aged 48 hours @ 100°C
3. Zwick Rotary Drum Abrader—ASTM D 5963, Method A
4. DeMattia crack growth—ASTM D813
5. Metravib DMA strain Sweep, shear mode, 10 Hz, 30 and 60°C; and Saw Powders, page 14

Executive summary

Globally, more than 30 billion pounds of post-consumer and post-industrial rubber materials are generated each year. The great majority of this material is generated by the one billion annual global tire removals; however, millions of pounds of post-industrial rubber are recycled primarily into turf and playground applications. Approximately 50 percent of the total available rubber material is burned for energy, limiting the capture of the full value of this resource. Reuse of materials in tire and rubber formulas captures value where this use can be proven as truly sustainable.

The use of micronized rubber powder in tire tread compounds reduces costs by up to 50 percent per unit and is “green for free”—generating oil, energy and CO2 savings. Used by most major tire companies in the world, micronized rubber powder is a well-established standard raw material in the industry.

In addition to the tire-based micronized rubber, other industrial polymers including NBR and EPDM are available as micronized rubber powder for potential use in hose applications. As with the tire industry, micronized rubber is a cost savings driver for other applications, and similarly provides risk mitigation as a hedge against volatile oil prices.

This paper will exhibit data that indicates that PolyDyne micronized rubber powder can improve DeMattia crack growth dramatically with increasing loadings up to 10 percent over the base compound formulation. This finding extends the possible application of micronized rubber powder into a myriad of applications, including hose and belt products, where resistance to crack growth and improved fatigue properties are required. A plausible hypothesis will be presented on the mechanism for improved crack growth performance when introducing micronized rubber powder in rubber compounds, which is supported by microscopic evidence. Information on the processing, physical properties and dynamic properties will be presented related to the use of micronized rubber powder in rubber compounds.

Table 1. Model compound recipe and PolyDyne formulations, with and without countermeasures.

<table>
<thead>
<tr>
<th>Rubber compound</th>
<th>Control</th>
<th>5% PolyDyne®</th>
<th>10% PolyDyne®</th>
<th>5% PD140 w/CB+S.</th>
<th>10% PD140 w/CB+S.</th>
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<tr>
<td></td>
<td>PPR</td>
<td>PPR</td>
<td>PPR</td>
<td>PPR</td>
<td>PPR</td>
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<tr>
<td>Natural Rubber SMR-1</td>
<td>40.00</td>
<td>40.00</td>
<td>40.00</td>
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<tr>
<td>High cis PBR (CB24)</td>
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<td>60.00</td>
<td>60.00</td>
<td>60.00</td>
<td>60.00</td>
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<tr>
<td>PBR, 140,200</td>
<td>10.15</td>
<td>21.42</td>
<td>10.16</td>
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<td>Sandex 8000</td>
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<tr>
<td>N550</td>
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<td>60.00</td>
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<td>SPPD Antidegradant</td>
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<td>Wax Blend</td>
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<td>Zinc Oxide Dispersion</td>
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<tr>
<td>Stearic Acid</td>
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<tr>
<td>CBS</td>
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<tr>
<td>Sulfur Dispersion</td>
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<td>2.13</td>
<td>2.39</td>
<td>2.66</td>
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<td>Retarder CTP</td>
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<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
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<tr>
<td>Total PPR</td>
<td>197.75</td>
<td>202.90</td>
<td>214.17</td>
<td>203.11</td>
<td>214.58</td>
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</table>
As measured by an MDR 2000 Rheometer minimum torque at 160°C, viscosity increases with increasing loading of MRP illustrated in Fig. 1. The finer particle size material, 200 mesh, typically minimizes this effect.

Scorch safety, Ts1, decreases slightly with increased loading of micronized rubber powder as shown in Fig. 2. Cure times, Tc90, also decrease slightly with increased loadings of micronized rubber powders yet are still manageable (Fig. 3).

Physical properties

Tensile properties were equivalent to control levels at 5 percent loadings of MRP and slightly reduced at 10 percent loadings. No differential effects were observed on aged tensile compared to control levels. Figs. 4 and 5 show these effects.

Modulus values of 80 and 140 mesh materials were similar but slightly lower than control values. Increased modulus was observed in 200 mesh. Use of counter measures had very little effect in unaged modulus yet lead to greater increase of modulus on aging. Figs. 6 and 7 illustrate these properties.

Elongation at break values were equivalent to control values at 5 percent loading for all micronized rubber powder sizes. At 10 percent loadings, elongation at break values were reduced for all MRP sizes. Counter measures exhibited similar values to the control for unaged specimens. No significant differential effects upon aging for all MRP sizes. Although when using counter measures, we observed slightly worse aging properties. Figs. 8 and 9 show these results.

Dynamic compound properties

Compounds containing 80 and 140 mesh materials exhibited a general increase in hysteresis as measured by tangent delta and 30°C.

The 200 mesh compound exhibited similar hysteresis levels relative to the control compound. No significant differences in storage modulus were observed for all PolyDyne containing compounds, including those with counter measures. Figs. 10 and 11 show the dynamic effects at 30°C.

Compounds containing PolyDyne 80 and 140 exhibited modest increases in hysteresis as measured by tangent delta and 60°C. PolyDyne 200 exhibited similar to lower hysteresis levels relative to the control compound.

No significant differences in Delta G’ were observed with MRP compounds, indicating similar micro-dispersion and filler-filler interactions.
**Figs. 12 and 13** show the dynamic effects at 60°C. Abrasion loss, measured by din abrasion, is similar for all PolyDyne containing compounds, including those with countermeasures vs. the control compound as illustrated in **Fig. 14**.

**Fatigue to failure and DeMattia crack growth results**

Improved fatigue to failure was observed by reducing particle size. Also reduced fatigue to failure was seen with increased loadings.

PolyDyne 140 and smaller particle size distribution are needed to match or improve crack initiation levels. **Fig. 15** illustrates these findings.

Significant reductions in crack growth rate for all PolyDyne containing compounds were observed. PolyDyne 140 with no countermeasures material might show a slightly lower crack growth rate compared with all other compounds. Crack growth improves with increased loading as shown in **Fig. 16**.

Crack growth is significantly improved with increased loadings of PolyDyne material. A slight preference toward smaller particle size is observed from the particle size effect graph (**Fig. 18**). PolyDyne 140 (105 µm) exhibits the greatest reduction in crack growth rate. **Figs. 17 and 18** illustrate the loading and particle size effects.

**Fig. 1.** Minimum torque as measured by MDR 2000.

**Fig. 2.** Scorch time, Ts1.

**Fig. 3.** Cure time, Tc90.

**Fig. 4.** Unaged tensile properties.

**Fig. 5.** Aged tensile properties.

**Fig. 6.** Unaged 300 percent modulus values.

**Fig. 7.** Aged 300 percent modulus values.

**Fig. 8.** Unaged elongation at break.

**Fig. 9.** Aged elongation at break.

**Fig. 10.** Tangent Delta at 30°C as measured by a DMA strain sweep at 10 Hz.

**Fig. 11.** Storage modulus at 30°C as measured by a DMA strain sweep at 10 Hz.

**Fig. 12.** Tangent Delta at 60°C as measured by a DMA strain sweep at 10 Hz.

**Fig. 13.** Delta G’ at 60°C as measured by a DMA strain sweep at 10 Hz.

**Fig. 14.** Abrasion loss as measured by din abrasion.

The improvements in crack growth performance are hypothesized to be related to the increase in fracture energy required when the crack is forced to deviate around the MRP particle which in turn would reduce the crack propagation rate. The mechanism is further supported by work done by S-C Han showing similar results in 100 percent NR and 100 percent SBR formulas.

**Figs. 19 and 20** exhibit this phenomenon showing crack growth in unfilled compounds (**Fig. 19**) and crack growth in MRP containing compounds (**Fig. 20**) as observed in optical microscopy.

In the control image (**Fig. 19**) one can observe very little crack diversion. In the 10 percent PolyDyne 140 image (**Fig. 20**) crack diversion around the particle, which potentially increases the fracture energy required to propagate the crack is observed.

**Conclusions**

During processing, typical effects on processing, cure and physical properties (both aged and unaged) were observed for all PolyDyne containing compounds. PolyDyne 200 containing materials exhibited the best balance of these properties.

See **Powder, page 16**.
Oil Eater pads.
For more information, visit www.oileater.com.

The Sarlink ME-2200 Series exhibit higher flow than comparable TPVs, Teknor Apex said, enabling molders of exterior components such as gaskets, seals and trim to process complex designs while shortening cycles through reduced packing and cooling time.

The company said the series provides a cost savings while meeting performance requirements of the part. Like TPVs, the Sarlink ME-2200 series compounds are less dense than EPDM and PVC, Teknor Apex claimed, yielding weight savings of up to 15 and 23 percent, respectively.

For more information, visit www.teknorapex.com.

Oil Eater, a brand of Kafko International Ltd., has introduced sonic bonded pads for a variety of spills in manufacturing facilities, including oils, coolants, solvents, water and acids. The pads and rolls tear easily along their perforated seam to fit almost any space, Oil Eater said, especially leaks in hard-to-reach places.

The sonic bonded pads are constructed from a single layer of high-quality uniform polypropylene fibers that have been bonded together using a unique high-loft process, the company said. Oil Eater said the material provides superior strength and reduced linting.

The line is available in a variety of weights in sizes from 15-inch by 18-inch pads to 30-inch by 150-foot rolls. A sample is available upon request. For more information, visit www.oileater.com.

The Association for Hose and Accessories Distribution has released its latest version of the Hose Safety Institute Handbook. An update pack is available for any existing copies.

The handbook is 324 pages and includes 93 charts and 319 photos. The association said images serve as an excellent reference, training and specification resource for hose assembly fabricators and end-users. The handbook can serve as a study guide for five different online exams, the group said.

NAHAD noted that previous versions of the handbook are no longer valid and are not supported by the institute. Copies are on sale at www.nahad.org.

References
2. Papp, F. “Optimizing the use of micronized rubber powder made from end-of-life tire material.” Rubber World 246 August 2012: 16-27

One unexpected result was the significant improvement in DeMattia crack growth observed for all PolyDyne containing materials as well as the improvement in DeMattia crack growth with increasing PolyDyne loadings. PolyDyne 140 containing materials with no counter measures appears to show the best crack growth performance compared to all other compounds studied.

A very plausible explanation for the decrease in crack growth rates for PolyDyne containing materials would be the crack diversion around the particles could increase the fracture energy required for crack propagation lowering the crack growth rate. This was observed via optical microscopy.

Future studies
We have several future studies in the works to help better understand this observed phenomenon of reduced crack growth rates. Currently, we are investigating the optimal particle size.

We are repeating the testing with specific particle size materials, a 40/60 cut, an 80/140 cut, a 140/200 cut, and a 200/300 cut of material to help determine optimal particle size.

A design of experiment (DOE) was used with these materials to vary the loading and particle size in each compound. Flex fatigue, DeMattia with and without precut for crack initiation, dynamic and static ozone crack testing will all be investigated in this DOE.