DRAGONITE™

Reinforcement, faster processing and fire-retardance using Halloysite a natural, tubular mineral

Presented by: Dr. Chris DeArmitt – CTO
SPE New Jersey Section 2011
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Agenda

- Introduction to fillers
- What is Halloysite?
- Mechanical properties
- Processing advantages
- Flame retardance
- Sustained release of actives
- Availability and pricing
- Conclusions
Halloysite Property Overview

- Aluminosilicate mineral: $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4 \cdot n\text{H}_2\text{O}$
- Molecular weight: 294.19
- CAS: 1332-58-7
- Density: $2.70 \pm 0.03 \text{gcm}^{-3}$
- Refractive index at room temperature: 1.534, dried at 100°C 1.548
- Specific heat capacity: 0.92 kJkg$^{-1}$K$^{-1}$
- Thermal conductivity: 0.092 WK$^{-1}$m$^{-1}$
- Thermal diffusivity: $5.04 \times 10^{-4}$ cm$^2$ sec$^{-1}$
- CTE: $10.0 \pm 1.5$ perpendicular to the layer, $6.0 \pm 2.0$ parallel
- Colorless and UV transparent
- pH in water 6.4
- Particle shape: 1-2 microns long, 50nm across, 15nm diameter hole
- Surface area: 65-120 m$^2$g$^{-1}$
- Dragonite™ purity: 95-100%
Halloysite is a natural aluminosilicate clay with a hollow tubular morphology.

Naturally exfoliated morphology means no need to chemically separate particles and makes for easy dispersion.

Halloysite nanotubes typically have diameter ~50nm with lengths ranging from 1 to 2 microns giving an aspect ratio of ~20.

Traditional uses include fine china, fillers in paints and paper, food extenders, catalysts and molecular sieves.
Dragonite™ Intrinsic Properties and Applications

High aspect ratio
- Reinforcement of plastics, elastomers, coatings etc.

High surface area
- Catalysts, adsorbents, carrier, elastomers, immobilization, nucleation of crystal growth and foam cell formation

Hollow
- Controlled release, thermal insulation, light-weighting, wicking, membranes, reverse osmosis

Bound water
- Fire retardance, temperature indicator, foaming agent
Markets Addressed

- **Plastics**
  - Productivity +20%
  - Mechanics +20%
  - Flame retardance

- **Environment**
  - Oil clean-up
  - Soil remediation
  - Water purification

- **Coatings & Adhesives**
  - UV cure speed +20%
  - Mechanics +20%
  - Improved adhesion

- **Elastomers**
  - Reinforcement
  - Flame retardance
  - Thermal stability
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# Dragonite in Plastics

<table>
<thead>
<tr>
<th>Plastic Type</th>
<th>Mechanical Reinforcement</th>
<th>Nucleation/ Cycle Time Reduction</th>
<th>Clear Film</th>
<th>Flame Retardance</th>
</tr>
</thead>
<tbody>
<tr>
<td>PE</td>
<td>✔</td>
<td>✔</td>
<td>✔</td>
<td>✔</td>
</tr>
<tr>
<td>PP</td>
<td>✔</td>
<td>✔</td>
<td>✔</td>
<td>✔</td>
</tr>
<tr>
<td>EVA</td>
<td>✔</td>
<td>TBD</td>
<td>✔</td>
<td>✔</td>
</tr>
<tr>
<td>PA6</td>
<td>✔</td>
<td>TBD</td>
<td>TBD</td>
<td>✔</td>
</tr>
<tr>
<td>PA12</td>
<td>✔</td>
<td>✔</td>
<td>TBD</td>
<td>✔</td>
</tr>
<tr>
<td>PVC</td>
<td>✔</td>
<td>TBD</td>
<td>✔</td>
<td>TBD</td>
</tr>
<tr>
<td>PLA</td>
<td>✔</td>
<td>✔</td>
<td>✔</td>
<td>TBD</td>
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<tr>
<td>Epoxy</td>
<td>✔</td>
<td>TBD</td>
<td>TBD</td>
<td>✔</td>
</tr>
<tr>
<td>EPDM</td>
<td>✔</td>
<td>TBD</td>
<td>TBD</td>
<td>✔</td>
</tr>
</tbody>
</table>

**Suggested Grade**
- Dragonite™ XR or HP
- Dragonite™ HP
- Dragonite™ HP
- Dragonite™ XR
Dispersibility of Halloysite and Polarity

- Halloysite has been shown to disperse well in all types of system, from apolar to very polar.
- Wetting through the tubes gives mechanical bonding even in cases where no specific chemical interaction takes place.
- In thermosets, thermoplastics and elastomers, effective reinforcement is reported even without dispersants or coupling agents.
- Dispersants and coupling agents may also be used.
Isotropic fillers retain impact but do not reinforce

- Reinforcing fillers ruin impact resistance and elongation to break
- Halloysite reinforces and retains or improves impact and elongation
- This is possible due to shape, surface area and easy dispersibility
## Halloysite in LLDPE

<table>
<thead>
<tr>
<th>Property</th>
<th>LLDPE</th>
<th>10% Halloysite</th>
<th>20% Halloysite</th>
<th>30% Halloysite</th>
<th>40% Halloysite</th>
<th>60% Halloysite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flexural Modulus (MPa)</td>
<td>180</td>
<td>327</td>
<td>453</td>
<td>570</td>
<td>635</td>
<td>1378</td>
</tr>
<tr>
<td>Flexural Strength (MPa)</td>
<td>8.2</td>
<td>12.7</td>
<td>14.9</td>
<td>16.8</td>
<td>19.1</td>
<td>23.0</td>
</tr>
<tr>
<td>Impact Resistance (KJm⁻²)</td>
<td>50</td>
<td>37.5</td>
<td>22.1</td>
<td>14.4</td>
<td>10.6</td>
<td>5.4</td>
</tr>
<tr>
<td>PHRR (KWm⁻²)</td>
<td>920</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>480</td>
<td>---</td>
</tr>
</tbody>
</table>

Strength and PHRR further increased by addition of graft copolymer coupling agent

Reinforcing and Flame-Retardant Effects of Halloysite Nanotubes on LLDPE
## Dragonite in PP Film

<table>
<thead>
<tr>
<th>Property</th>
<th>PP</th>
<th>1% Dragonite</th>
<th>2% Dragonite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Modulus (psi)</td>
<td>129</td>
<td>140</td>
<td>143</td>
</tr>
<tr>
<td>Yield Strength (psi)</td>
<td>4046</td>
<td>4978</td>
<td>5379</td>
</tr>
<tr>
<td>Tear Strength</td>
<td>1178</td>
<td>1142</td>
<td>1122</td>
</tr>
<tr>
<td>Elongation to break</td>
<td>540</td>
<td>610</td>
<td>574</td>
</tr>
</tbody>
</table>

When blowing film an increase in bubble stability was noted.
## Dragonite in PVC

<table>
<thead>
<tr>
<th>Property</th>
<th>PVC</th>
<th>1% Dragonite</th>
<th>2% Dragonite</th>
<th>3% Dragonite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Modulus (kpsi)</td>
<td>459</td>
<td>490</td>
<td>488</td>
<td>521</td>
</tr>
<tr>
<td>Yield Strength (psi)</td>
<td>13084</td>
<td>13771</td>
<td>13834</td>
<td>14010</td>
</tr>
<tr>
<td>Elongation to break (%)</td>
<td>6</td>
<td>10</td>
<td>9</td>
<td>8</td>
</tr>
<tr>
<td>Notched Izod (ft-lb/in)</td>
<td>0.9</td>
<td>0.9</td>
<td>0.9</td>
<td>0.8</td>
</tr>
<tr>
<td>Unnotched Izod (ft-lb/in)</td>
<td>7.3</td>
<td>7.2</td>
<td>10.2</td>
<td>12.5</td>
</tr>
</tbody>
</table>
# Dragonite in PA6

<table>
<thead>
<tr>
<th>Property</th>
<th>PA6</th>
<th>4% Dragonite</th>
<th>8% Dragonite</th>
<th>14% Dragonite</th>
<th>19% Dragonite</th>
<th>27% Dragonite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flexural Modulus (Kpsi)</td>
<td>398</td>
<td>521</td>
<td>539</td>
<td>671</td>
<td>700</td>
<td>891</td>
</tr>
<tr>
<td>Flexural Strength (psi)</td>
<td>14785</td>
<td>18428</td>
<td>18351</td>
<td>20272</td>
<td>21041</td>
<td>21817</td>
</tr>
<tr>
<td>Notched Izod Impact (ft-lb/inch)</td>
<td>1.2</td>
<td>0.94</td>
<td>0.90</td>
<td>0.95</td>
<td>0.94</td>
<td>0.93</td>
</tr>
<tr>
<td>Density (g/cm³)</td>
<td>1.13</td>
<td>1.16</td>
<td>1.186</td>
<td>1.226</td>
<td>1.262</td>
<td>1.317</td>
</tr>
</tbody>
</table>

Dragonite pre-dried, no surface treatment
Case Study #1
Dragonite HP™ in HDPE Temporary Flooring

Objective:
• To increase strength and stiffness of a 1000lb compression molded part while retaining the excellent impact resistance of HDPE
• Reduce warpage caused by thermal expansion and contraction (CTE)

Solution:
• Reduced warpage & CTE by with as little as 1% loading of Dragonite HP™
• Retained the impact resistance of the control
• Drop-in solution
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Case Study #2
Cycle Time Reduction for HDPE Part

PHASE 1:
Drop-in solution: Significant cycle time reduction

<table>
<thead>
<tr>
<th></th>
<th>Virgin HDPE</th>
<th>HDPE + 1% Dragonite HP</th>
<th>Savings</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cycle time per part (seconds)</td>
<td>107</td>
<td>80</td>
<td>25%</td>
</tr>
<tr>
<td>Parts per hour</td>
<td>34</td>
<td>45</td>
<td>32%</td>
</tr>
<tr>
<td>Cost per part ($)</td>
<td>8.07</td>
<td>7.53</td>
<td>7%</td>
</tr>
</tbody>
</table>

Effective cost of HDPE ($/lb) 0.85 0.76 11%

- At 1 wt% Dragonite-HP loading, the customer achieved a 25% reduction in cycle time resulting in significant manufacturing cost reduction
- Results based on actual commercial process of the end user
- The customer was able to reduce the visibility of sink marks by >60%
- A 20% increase in stiffness without affecting impact resistance of the final part
- Also validated in PP copolymer and homopolymer
- Applies to injection molding and extrusion

PHASE 2: Testing in Progress
Additional savings through thin-walling

Better mechanicals enables thin-walling:
- 5-10% reduction in wall thickness
- 10% further reduction in cycle time
- 5–10¢ per lb additional savings
Low Shear Viscosity in PP (230°C)

![Graph showing viscosity vs. shear rate for different PP modifications.]

- **Unmodified PP**
- **PP +1% Dragonite**
- **PP +25% Dragonite**

Viscosity (Pa.s) vs. Shear Rate (1/s)
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## Flame Retardancy:
**Dragonite-XR vs. MDH- Magnesium Hydroxide**

<table>
<thead>
<tr>
<th></th>
<th>Control</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dragonite XR</td>
<td>0</td>
<td>0</td>
<td>20</td>
<td>30</td>
<td>40</td>
<td>60</td>
</tr>
<tr>
<td>Magnesium Hydroxide (ST)</td>
<td>0</td>
<td>60</td>
<td>40</td>
<td>30</td>
<td>20</td>
<td>0</td>
</tr>
<tr>
<td>PP 20 MFI</td>
<td>100</td>
<td>40</td>
<td>40</td>
<td>40</td>
<td>40</td>
<td>40</td>
</tr>
<tr>
<td>Flexural Modulus tangent (kpsi)</td>
<td>207</td>
<td>432</td>
<td>467</td>
<td>464</td>
<td>521</td>
<td>557</td>
</tr>
<tr>
<td>Flexural Modulus 1% (kpsi)</td>
<td>212</td>
<td>373</td>
<td>391</td>
<td>392</td>
<td>440</td>
<td>461</td>
</tr>
<tr>
<td>Flexural Strength (psi)</td>
<td>6517</td>
<td>5131</td>
<td>5350</td>
<td>5347</td>
<td>5666</td>
<td>6200</td>
</tr>
<tr>
<td>Tensile Modulus (kpsi)</td>
<td>150</td>
<td>277</td>
<td>275</td>
<td>285</td>
<td>300</td>
<td>294</td>
</tr>
<tr>
<td>Tensile Strength (psi)</td>
<td>5180</td>
<td>3242</td>
<td>3182</td>
<td>3189</td>
<td>3650</td>
<td>3818</td>
</tr>
<tr>
<td>Notched Izod Impact ft-lb/in</td>
<td>0.44</td>
<td>0.54</td>
<td>0.54</td>
<td>0.5</td>
<td>0.45</td>
<td>0.43</td>
</tr>
<tr>
<td>Smoke</td>
<td>low</td>
<td>low</td>
<td>very low</td>
<td>very low</td>
<td>low</td>
<td></td>
</tr>
<tr>
<td>UL 94 Rating</td>
<td>V2</td>
<td>V1</td>
<td>V1</td>
<td>V1</td>
<td>V1</td>
<td></td>
</tr>
</tbody>
</table>
Advantages of Dragonite-XR™
Reduced Flammability

Heat release rate of neat PP and PP/Halloysite composites

Source: 2010 Society of Chemical Industry: Du, Guo, Jia
PET FR Development

- Reinforcing, halogen free flame retardant
- Good mechanics in combination with glass fiber
- High water release temperature >400°C means Dragonite™ is ideally suited to polymers processed at high temperature
- Char strength boosted with Dragonite-XR plus glass fiber
- Synergistic fluxing effect under investigation

30% + 5% GF  30% + 10% GF  30% + 15% GF
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Halloysite Tubules
Controlled-Release

- 0.5 nm
- 7 nm
- 15 nm

Glucose
180 Da

Hemoglobin Protein
60 kDa

Dragonite™
Loading the Tubes

<table>
<thead>
<tr>
<th>Property</th>
<th>Density</th>
<th>Weight % Oil</th>
<th>Volume % Oil</th>
<th>Loading %*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dragonite</td>
<td>2.666</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>1 Wintergreen</td>
<td>2.503</td>
<td>5.2</td>
<td>11</td>
<td>58</td>
</tr>
<tr>
<td>2 Wintergreen</td>
<td>2.583</td>
<td>2.7</td>
<td>5.9</td>
<td>31</td>
</tr>
<tr>
<td>3 Wintergreen</td>
<td>2.111</td>
<td>21</td>
<td>37</td>
<td>190</td>
</tr>
<tr>
<td>4 Winter &amp; Cedar</td>
<td>2.479</td>
<td>5</td>
<td>12</td>
<td>63</td>
</tr>
<tr>
<td>5 Tinuvin 292</td>
<td>2.467</td>
<td>5.2</td>
<td>13</td>
<td>68</td>
</tr>
<tr>
<td>6 Tinuvin 292</td>
<td>2.448</td>
<td>5</td>
<td>13</td>
<td>68</td>
</tr>
<tr>
<td>7 Tinuvin 292</td>
<td>2.466</td>
<td>5.2</td>
<td>13</td>
<td>68</td>
</tr>
<tr>
<td>8 Mustard</td>
<td>2.620</td>
<td>1</td>
<td>2.6</td>
<td>14</td>
</tr>
</tbody>
</table>

Based on Halloysite having 19% Lumen volume
Cedar Oil 0.95 gcm⁻³ Oil of Wintergreen 1.17 gcm⁻³ Tinuvin 292: 0.99 gcm⁻³
50:50 Cedar + Wintergreen assumed 1.06 gcm⁻³ Mustard 1.00 gcm⁻³
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Nanotubes in Perspective

...the production capacity for all carbon nanotubes, nanofibers, graphenes, fullerenes and nanodiamonds was 4,065 tons in 2010, and is expected to exceed 12,300 tons in 2015. The actual production was less than 25% of the capacity in 2010 and about 50% of the capacity in 2015. Total production value is estimated at about $435 million in 2010 and is expected reach a value of $1.3 billion in 2015.

- Production capacity of the Dragon Mine exceeds the global capacity of all those materials combined

- Furthermore, Dragonite is safe, natural and is less than 1/100\textsuperscript{th} the cost of even the cheapest carbon nanotubes
Availability and Pricing

- Dragonite™ brand high-purity Halloysite is commercially available from Applied Minerals
- Dragonite™ is shipped directly from the Dragon Mine in Utah, USA
- Masterbatch concentrates are available as well as neat powder
- Supply is plentiful (>30ktons) to support large-scale applications
- Pricing is in the $1-3 / lb range
- Samples are available to interested parties
- Technical support is also available
Conclusions

- Nanotubular materials have long held great promise
- High cost, lack of availability and other factors have slowed progress until now
- Dragonite™ is 100% natural, safe, cost-effective and abundant enough to support large-scale commercial applications
- Due to high aspect ratio, surface area and easy dispersibility, Halloysite provides effective reinforcement to plastics and elastomers
- Water release at high temperature give a halogen-free FR alternative for high temperature and engineering polymers
- The hollow tubes provide a controlled release effect
Thank You For Your Time

Q&A
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