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A Leilani Richardson (661) 275-5015

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Prescribed by ANSI Std. 239.18

2 items enclosed = 212 + 211
“Effects on Processing by Drop-in Modifiers in Nano-Composite Polymers”

Patrick Ruth,
Senior Technician, AFRL/PRSM
Air Force Research Lab, Edwards

Brent Viers, Rusty Blanski, and Andre Lee

MEMORANDUM FOR PRS (In-House Contractor Publication)

FROM: PROI (STINFO) 03 Sept 2002


Patrick Ruth (ERC) et al., “Effects on Processing by Drop-in Modifiers in Nano-Composite Polymers” (viewgraphs)

POSS Nanotechnology Conference
(Huntington Beach, CA, 25-27 September 2002) (Deadline: 25 Sept 02) (Statement A)
POSS As a Drop-in Modifier- Introduction

What is POSS? (Simplified)
1. Structure
2. Functional Groups and Dropping-in
3. Proposed and Actual uses

Making Samples
1. Material Selection and Preparation
2. Blending
3. Sample Production
Anatomy of a Polyhedral Oligomeric Silsesquioxane (POSS™) Molecule

Nonreactive organic (R) groups for solubilization and compatibilization.

Nanoscopic in size with an Si-Si distance of 0.5 nm and a R-R distance of 1.5 nm.

May possess one or more functional groups suitable for polymerization or grafting.

Thermally and chemically robust hybrid (organic-inorganic) framework.

Precise three-dimensional structure for molecular level reinforcement of polymer segments and coils.
POSS Chemically Incorporated into Plastics

POSS-Kapton

POSS-EPOXY

POSS-PMMA
POSS Blended into Plastics
Materials Selection: Polypropylene and POSS

atactic polypropylene

syndiotactic polypropylene

isotactic Polypropylene

Methyl$_8$T$_8$
i-PP/Me₈T₈ Processing Studies

iso-Polypropylene w/ Me8T8

Load (Newtons)

0.00  1.00  2.00  3.00

Time (min)

- - i-PP
- - - i-PP/10wt%Me8T8
- - - - i-PP/20wt%Me8T8
- - - - - i-PP/30wt%Me8T8
### Prof. Andre Lee - Michigan State University

<table>
<thead>
<tr>
<th></th>
<th>Dow data</th>
<th>Neat i-PP (processed)</th>
<th>i-PP blended 2 wt% Methyl₈T₈</th>
<th>i-PP blended 5 wt% Methyl₈T₈</th>
<th>i-PP blended 10 wt% Methyl₈T₈</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Tensile Strength @ Yield; ASTM D638</strong></td>
<td>5000 psi (34.5 MPa)</td>
<td>4800 psi (33.0 MPa)</td>
<td>5000 psi (34.5 MPa)</td>
<td>5100 psi (35.1 MPa)</td>
<td>5200 psi (35.8 MPa)</td>
</tr>
<tr>
<td><strong>Flexural Modulus (0.05 in/min, 1% secant); ASTM D790A</strong></td>
<td>240,000 psi (1.655 GPa)</td>
<td>235,000 psi (1.620 GPa)</td>
<td>251,000 psi (1.730 GPa)</td>
<td>255,000 psi (1.757 GPa)</td>
<td>262,000 psi (1.80 GPa)</td>
</tr>
<tr>
<td><strong>HDT @ 66 psi, as injected; ASTM D648</strong></td>
<td>210 °F (99 °C)</td>
<td>210 °F (99 °C)</td>
<td>221 °F (105 °C)</td>
<td>239 °F (115 °C)</td>
<td>255 °F (124 °C)</td>
</tr>
<tr>
<td><strong>Impact Izod @25C ASTM D256A</strong></td>
<td>0.5 ft-lb/in</td>
<td>0.55 ft-lb/in</td>
<td>0.55 ft-lb/in</td>
<td>0.62 ft-lb/in</td>
<td>0.75 ft-lb/in</td>
</tr>
</tbody>
</table>

- The above data (other than Dow's data) is an average of at least 10 samples for each test with acceptable S.D. of 5% or better.
Polymer Processing Lab
Polymer Processing Parameters

- Time (10 Min)
- Pressure (Varied)
- Temperature (216°C)
Procedure

- DSC (Establish processing and drying temperatures)
- Drying (Vacuum Oven)
- DACA (Mixing)
- Press (Forming samples)
- Tests to compare properties
Polypropylene DSC

- Crystallization
- Melting
DACA Twin-screw Extruder

Torque
Feed
Exit
Load
Recycle Valve
## DACA Twin Screw Processing Parameters for Me8T8/iPP nanocomposite blends.

<table>
<thead>
<tr>
<th>Mix #</th>
<th>PP</th>
<th>Me₈T₈</th>
<th>Load (N)</th>
<th>Torque (Nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Material Percentage</td>
<td>Mix Duration (min)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Dried</td>
<td>Not Dried</td>
<td>Dried</td>
<td>Not Dried</td>
</tr>
<tr>
<td>1</td>
<td>100</td>
<td>Dried</td>
<td>3500</td>
<td>3200</td>
</tr>
<tr>
<td>2</td>
<td>100</td>
<td>Not Dried</td>
<td>3500</td>
<td>3100</td>
</tr>
<tr>
<td>3</td>
<td>90</td>
<td>10</td>
<td>3200</td>
<td>3000</td>
</tr>
<tr>
<td>4</td>
<td>90</td>
<td>10</td>
<td>3200</td>
<td>3100</td>
</tr>
<tr>
<td>5</td>
<td>90</td>
<td>10</td>
<td>3500</td>
<td>3250</td>
</tr>
<tr>
<td>6</td>
<td>90</td>
<td>10</td>
<td>3400</td>
<td>3200</td>
</tr>
</tbody>
</table>
Load Comparison of Dried and Not Dried Polypropylene with Octa-Methyl POSS

Dried Polypropylene
Dried PP, Dried POSS
Dried PP, Not Dried POSS

Not Dried Polypropylene
Not Dried PP, Dried POSS
Not Dried PP, Not Dried POSS
Load Comparison of Dried and Not Dried Polypropylene with Octa-Methyl POSS

- Dried Polypropylene
- Not Dried Polypropylene
- Dried PP, Dried POSS
- Not Dried PP, Dried POSS
- Dried PP, Not Dried POSS
- Not Dried PP, Not Dried POSS
Torque Comparison of Dried and Not-Dried Polypropylene and Octa-Methyl POSS in DACA Twin-Screw Extruder

- Dried Polypropylene
- Dried PP, Dried POSS
- Dried PP, Not Dried POSS
- Not Dried Polypropylene
- Not Dried PP, Dried POSS
- Not Dried PP, Not Dried POSS
Polypropylene/Me₈t₈ Extrudates
Hot Press

4 X4 Inch Mold
Pressed Film of DACA Extruded POSS/PP Blend Variants

1 Not Dried PP
2 Dried PP
3 Dried PP, Dried POSS
4 Dried PP, Not Dried POSS
5 Not Dried PP, Dried POSS
6 Not Dried PP, Not Dried POSS
SUMMARY

Drying plays a role in making Me₈T₈ compatible with isotactic polypropylene

Load/torque to mix the polymer with the POSS is increased if either of the components is not dried.

Visually, the most compatible of the mixes is where both POSS and PP components were dried. The extruded rod and pressed thin film are nearly as clear as pure polypropylene in the melt.
ACKNOWLEDGEMENTS

AFRL/PRSM: Dr. Brent Viers, Dr. Rusty Blanski, and Dr. Andre Lee
Air Force Research Lab Polymer Working Group

Hybrid Plastics: Dr. Joe Lichtenhan, Dr. Joe Schwab, and
Mr. Michael J Carr

This talk is as much about me learning my work as it is making samples.
A great deal of thanks goes to the people who do similar work and have
shown me tricks to make the technician look clever.