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FROM: PROI (TI) (STINFO) 17 Apr 2000

Blanksi, R., Phillips, S., Chaffee, K.; Lichtenhan, J. (Hybrid Plastics); Lee, A. & Geng, H.P. (Michigan State University), "The Synthesis of Hybrid Materials by the Blending of Polyhedral Oligosilsesquioxanes into Organic Polymers"


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ROBERT C. CORLEY (Date)
Senior Scientist (Propulsion)
Propulsion Directorate
The Synthesis of Hybrid Materials by the Blending of Polyhedral Oligosilsesquioxanes into Organic Polymers

Rusty L. Blanski\textsuperscript{1}, Shawn H. Phillips\textsuperscript{1}, Kevin Chaffee\textsuperscript{1}, Joseph Lichtenhan\textsuperscript{2}, Andre Lee\textsuperscript{3}, and Hei Ping Geng\textsuperscript{3}.

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Hybrid Organic/Inorganic Blends

- GOAL: To study the interaction and solubility of Polyhedral Oligosilsesquioxane (POSS) molecules containing various organic side groups with the polymer matrix.
- Polystyrene was chosen since it is readily available and can easily be solvent cast with the POSS molecules for TEM studies.
Why Use Blendables?

- Easier to tailor the organic side groups of the POSS molecule to give a polymer-soluble species
- Simple blending techniques can be used instead of copolymerization with reactive POSS monomers
- Potential Drop-in molecular modifier without requiring expensive replacement of processing equipment
POSS = Polyhedral Oligomeric Silsesquioxane

General Synthesis

\[ \text{RSiCl}_3 + \text{H}_2\text{O} \]

\[ \text{THF, NEt}_3, \text{HCl} \]

R = cyclopentyl vinyl

R = cyclopentyl
POSS = Polyhedral Oligomeric Silsesquioxane
General Synthesis
Preparation of Styrene-POSS Blends

- TEM Method
- Dissolve the Styrene and POSS in THF
- Cast very thin film by slow solvent evaporation
- **Traditional Processing**
  - Place Polystyrene in Extruder
  - Add POSS
  - Blend 2-5 Minutes
POSS Blends - Crystal Formation

50 wt % Cp₈T₈ in 2 million mol. wt. Polystyrene

R = cyclopentyl

Cp₈T₈

TEM image clearly shows formation of immiscible POSS crystallites (20-50k molecules)
POSS Blends - Crystal Formation

50 wt % $V_i T_8$ in 2 million mol. wt. Polystyrene

$V_i T_8$

TEM image clearly shows immiscibility in polymer system
POSS Blends - Increased Solubility

50 wt % Cp₇T₈Styryl in 2 million mol. wt. Polystyrene

R = cyclopentyl

TEM image shows significant decrease in size of crystallites
POSS Blends - Miscibility

50 wt % Styrenyl$_8$T$_8$ in 2 million mol. wt. Polystyrene

- White domains represent pure polystyrene (process issue)
- Grey domains represent miscible POSS/polystyrene
- Black dots are POSS crystallites (<100 POSS molecules)
- 30% increase in surface hardness of the material
POSS Blends - Miscibility

50 wt % Phenethyl₈T₈ in 2 million mol. wt. Polystyrene

\[ R = \text{Phenethyl} \]

- Demonstrated Complete Miscibility!!
- Grey domains represent miscible POSS/polystyrene
- Black dots are POSS crystallites (<100 POSS molecules)
Conclusions

• The organic side groups on the POSS molecule are extremely important in determining the solubility of the POSS in polystyrene

• The addition of the more soluble styrenyl POSS into styrene leads to an increase in surface hardness without adversely affecting polymer properties

• POSS can be thought of as functionalized silicas with the side groups acting as solubility enhancers
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