

2303M1A3

MEMORANDUM FOR PR (Contractor/In-House Publication)

FROM: PROI (TI) (STINFO)

17 Apr 2000

SUBJECT: Authorization for Release of Technical Information, Control Number: **AFRL-PR-ED-TP-2000-070**
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"

Materials Research Society Meeting (Statement A)
(San Francisco, CA, 24 Apr - 02 May 2000) (Submission Deadline: 02 May 2000)

1. This request has been reviewed by the Foreign Disclosure Office for: a.) appropriateness of distribution statement, b.) military/national critical technology, c.) export controls or distribution restrictions, d.) appropriateness for release to a foreign nation, and e.) technical sensitivity and/or economic sensitivity.

Comments: _____

Signature _____ Date _____

2. This request has been reviewed by the Public Affairs Office for: a.) appropriateness for public release and/or b) possible higher headquarters review.

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3. This request has been reviewed by the STINFO for: a.) changes if approved as amended, b.) appropriateness of distribution statement, c.) military/national critical technology, d.) economic sensitivity, e.) parallel review completed if required, and f.) format and completion of meeting clearance form if required

Comments: _____

Signature _____ Date _____

4. This request has been reviewed by PR for: a.) technical accuracy, b.) appropriateness for audience, c.) appropriateness of distribution statement, d.) technical sensitivity and economic sensitivity, e.) military/national critical technology, and f.) data rights and patentability

Comments: _____

APPROVED/APPROVED AS AMENDED/DISAPPROVED

ROBERT C. CORLEY (Date)
Senior Scientist (Propulsion)
Propulsion Directorate



The Synthesis of Hybrid Materials by the Blending of Polyhedral Oligosilsesquioxanes into Organic Polymers

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Merced





Hybrid Organic/Inorganic Blends

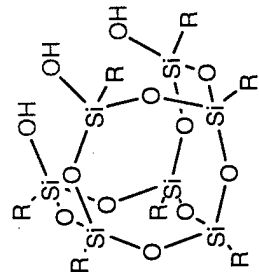
- **GOAL:** To study the interaction and solubility of Polyhedral Oligosilsesquioxane (POSS) molecules containing various organic side groups with the polymer matrix
- Polystyrene was chosen since it is readily available and can easily be solvent cast with the POSS molecules for TEM studies



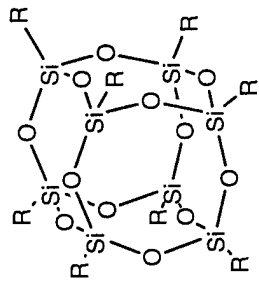
Why Use Blendables?

- 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

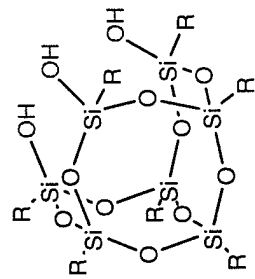
POSS = Polyhedral Oligomeric Silsesquioxane Synthesis



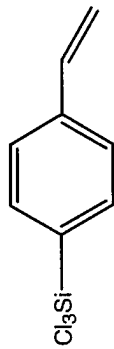
R = cyclopentyl
vinyl



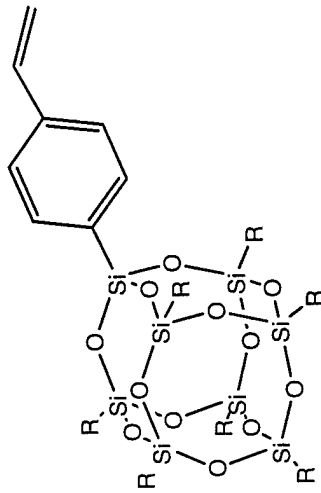
R = cyclopentyl
vinyl



R = cyclopentyl

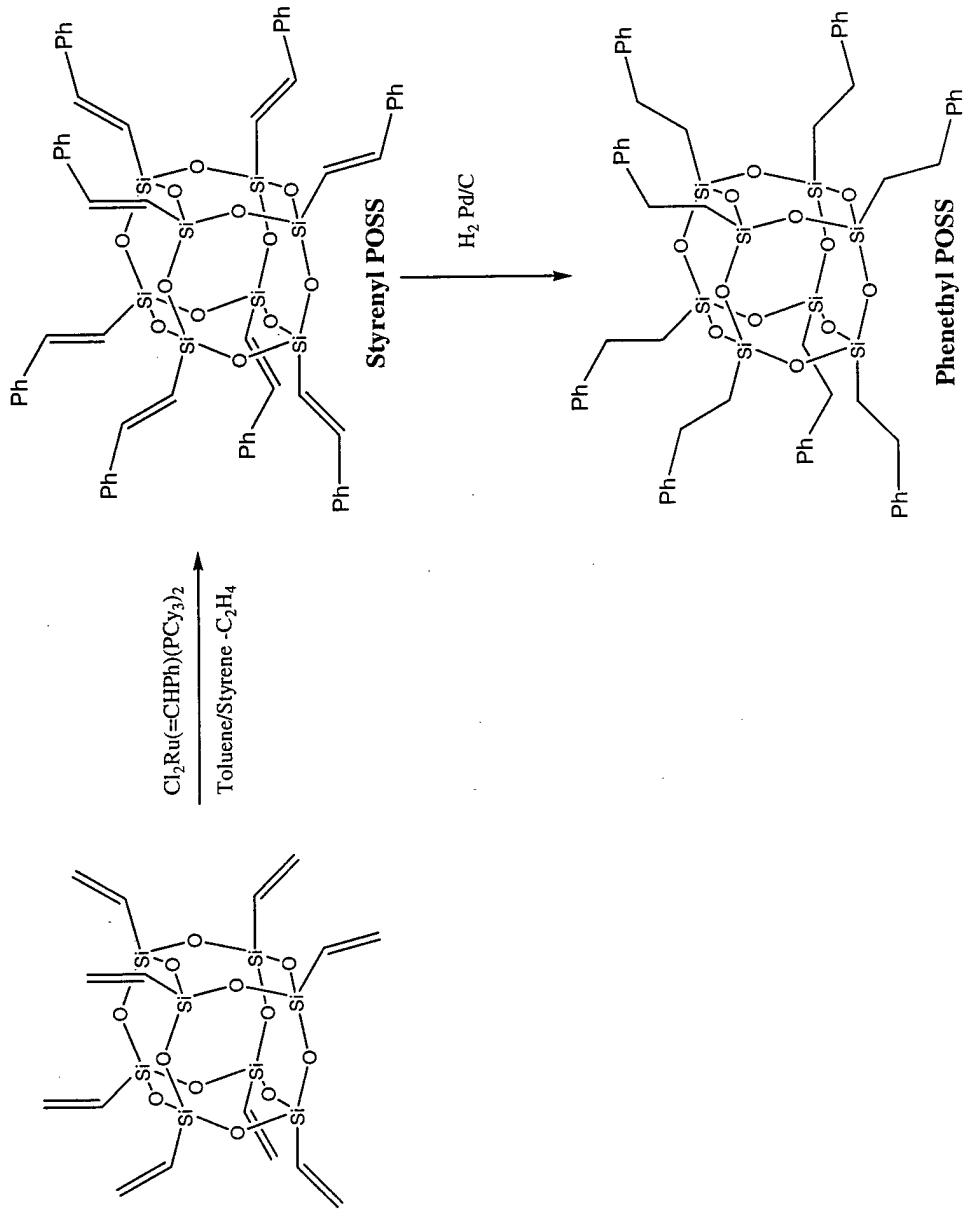


THF, NEt_3 , $\text{-NEt}_3 \cdot \text{HCl}$



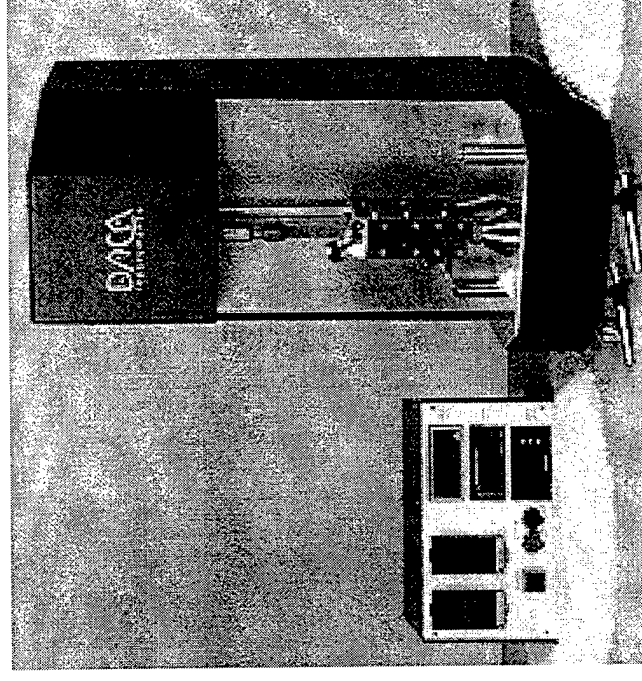
R = cyclopentyl

POSS = Polyhedral Oligomeric Silsesquioxane General Synthesis



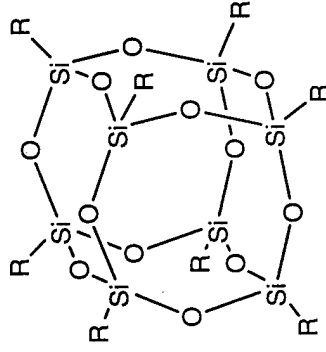
Preparation of Styrene-POSS Blends

- 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



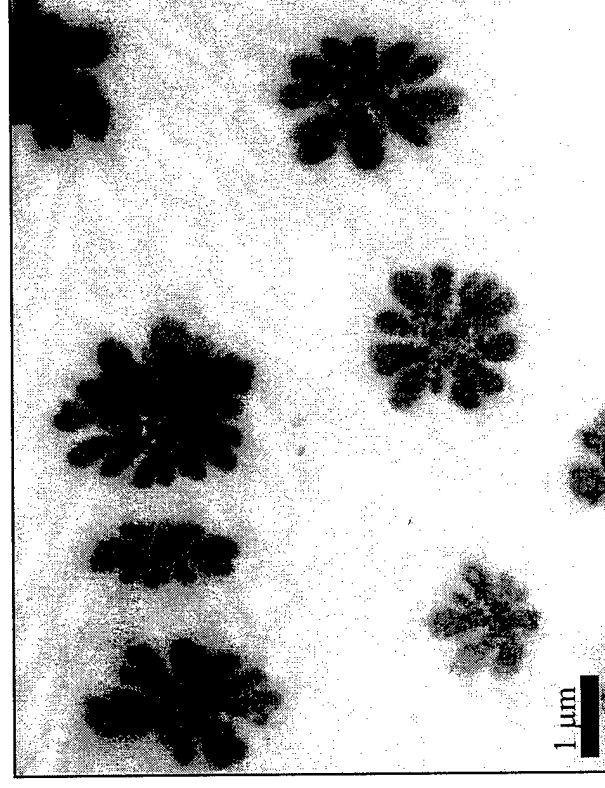
POSS Blends - Crystal Formation

50 wt % Cp_8T_8 in 2 million mol. wt. Polystyrene



R = cyclopentyl

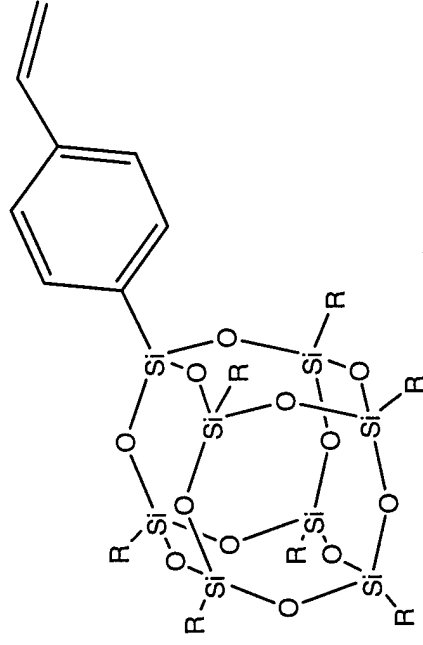
Cp_8T_8



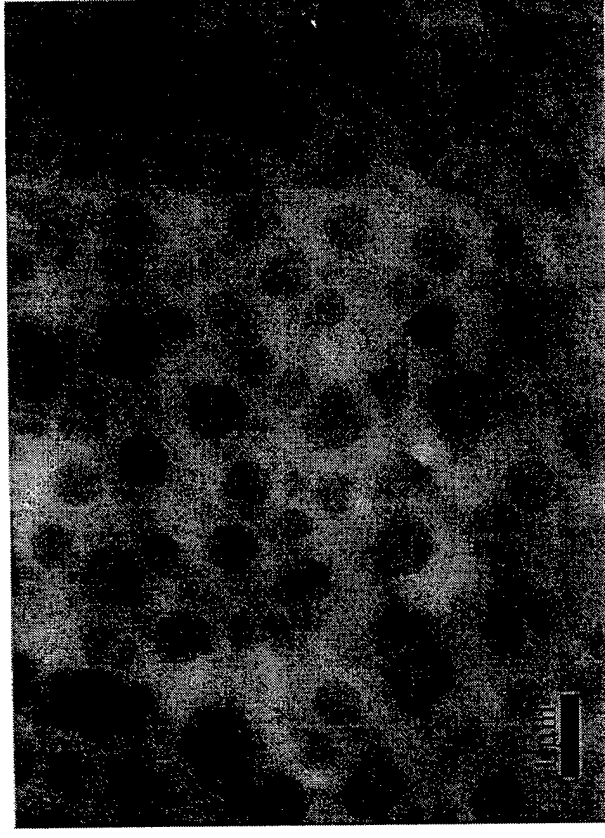
TEM image clearly shows formation of immiscible POSS crystallites (20-50k molecules)

POSS Blends - Increased Solubility

50 wt % Cp₇T₈ Styryl in 2 million mol. wt. Polystyrene



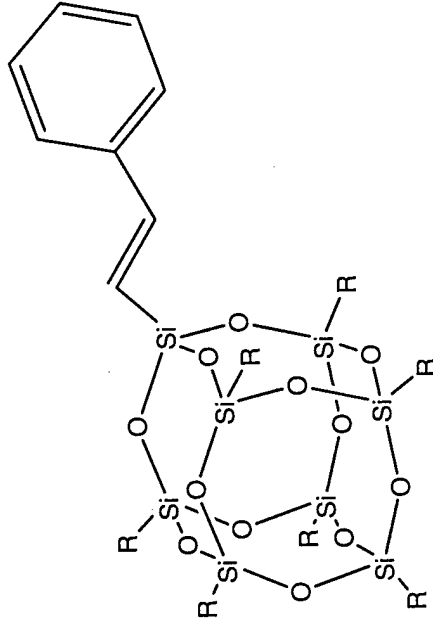
R = cyclopentyl



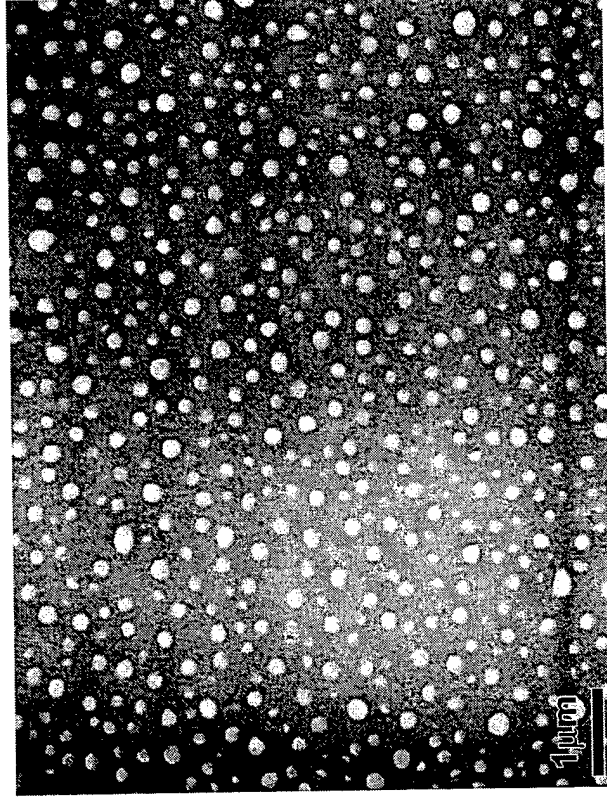
TEM image shows significant decrease in size of crystallites

POSS Blends - Miscibility

50 wt % Styrenyl₈T₈ in 2 million mol. wt. Polystyrene



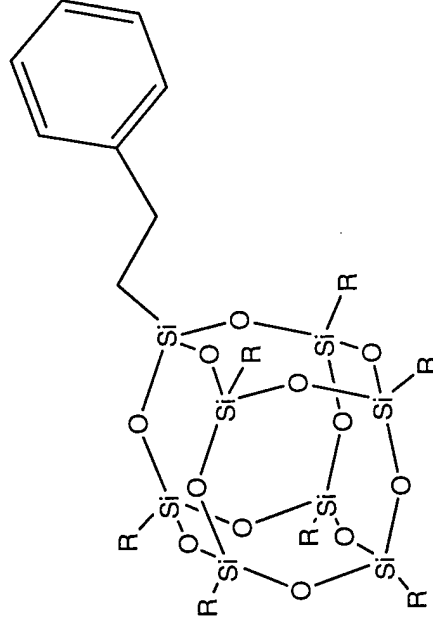
R = Styrenyl



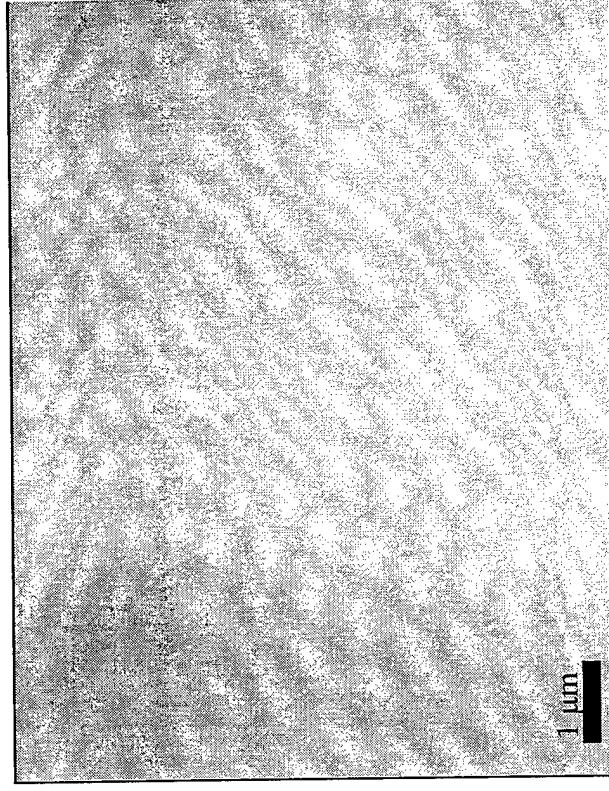
- White domains represent pure polystyrene (process issue)
- Grey domains represent miscible POSS/polystyrene
- Black dots are POSS crystallites (<100 POSS molecules)
- 30% increase in surface hardness of the material

POSS Blends - Miscibility

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



R = 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



Acknowledgements

- AFRL Propulsion Directorate
- Mr. Paul Jones (Analytical)
- Dr. Charles Lee, AFOSR (Funding)