

REPORT DOCUMENTATION PAGE

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MEMORANDUM FOR PRS (In-House Publication)

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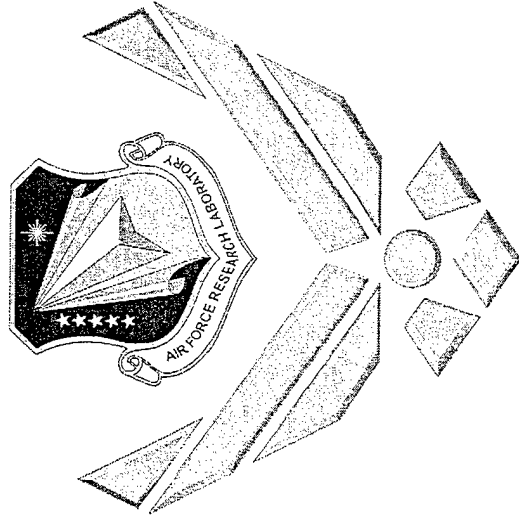
16 April 2002

SUBJECT: Authorization for Release of Technical Information, Control Number: **AFRL-PR-ED-VG-2002-083**
Patrick Ruth et al. (PRSM), "Effects on Processing by Drop-in Modifiers in Nano-Composite Polymers"

SAMPE Industry Conference
(Long Beach, CA, 12-15 May 2002) (Deadline: 12 May 2002)

(Statement A)

*“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

DISTRIBUTION STATEMENT A
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POSS as a Drop-in Modifier- Introduction

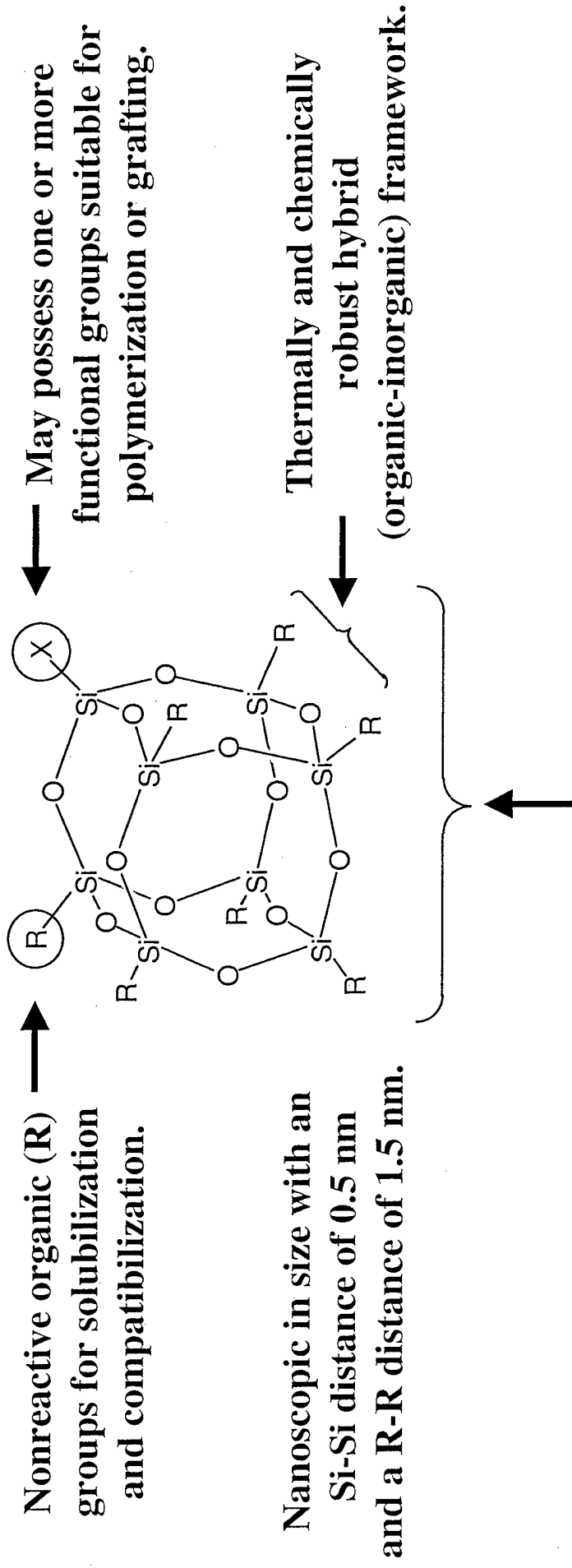
What is POSS? (Simplified)

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

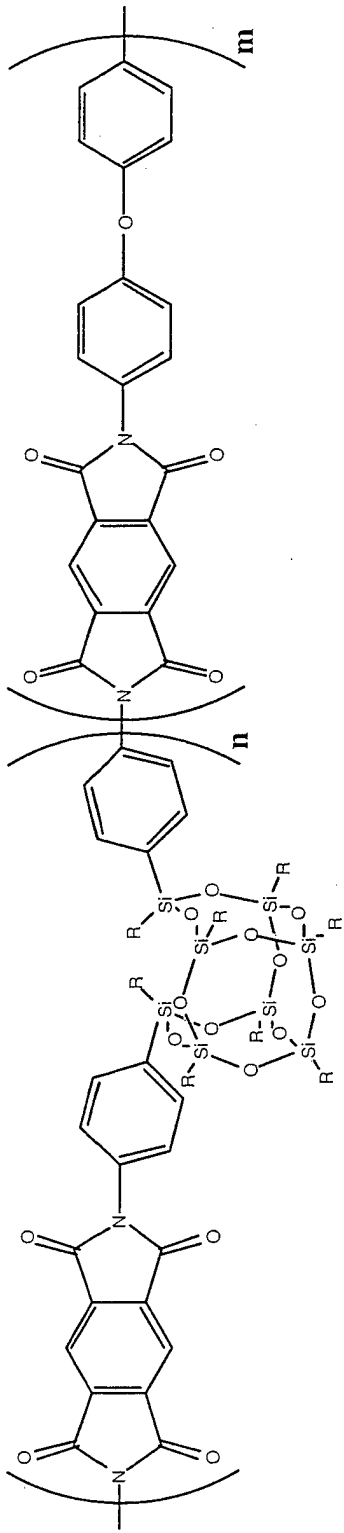
Anatomy of a Polyhedral Oligomeric Silsesquioxane (POSS™) Molecule



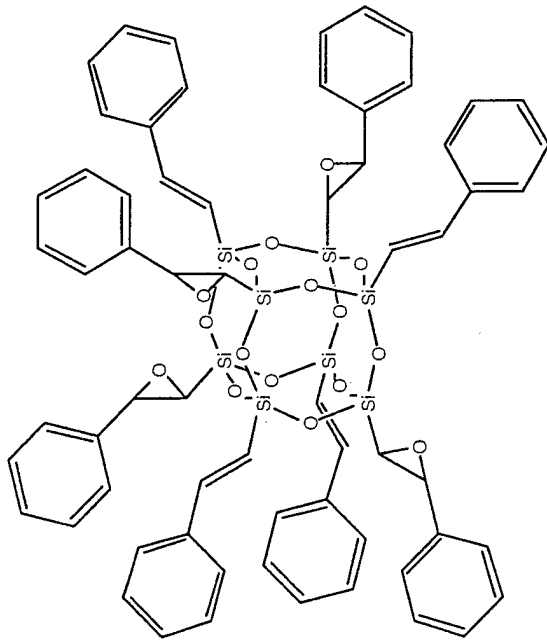
Nanosopic in size with an Si-Si distance of 0.5 nm and a R-R distance of 1.5 nm.

Precise three-dimensional structure for molecular level reinforcement of polymer segments and coils.

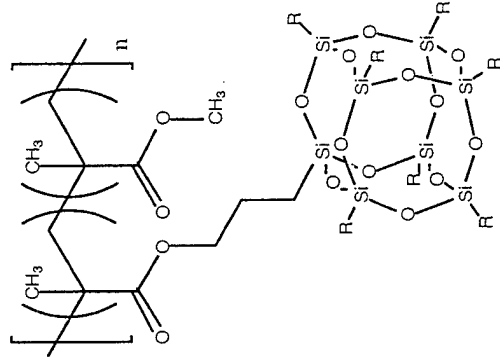
POSS Chemically Incorporated into Plastics



POSS-Kapton

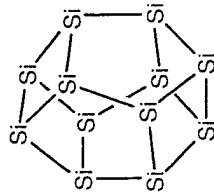
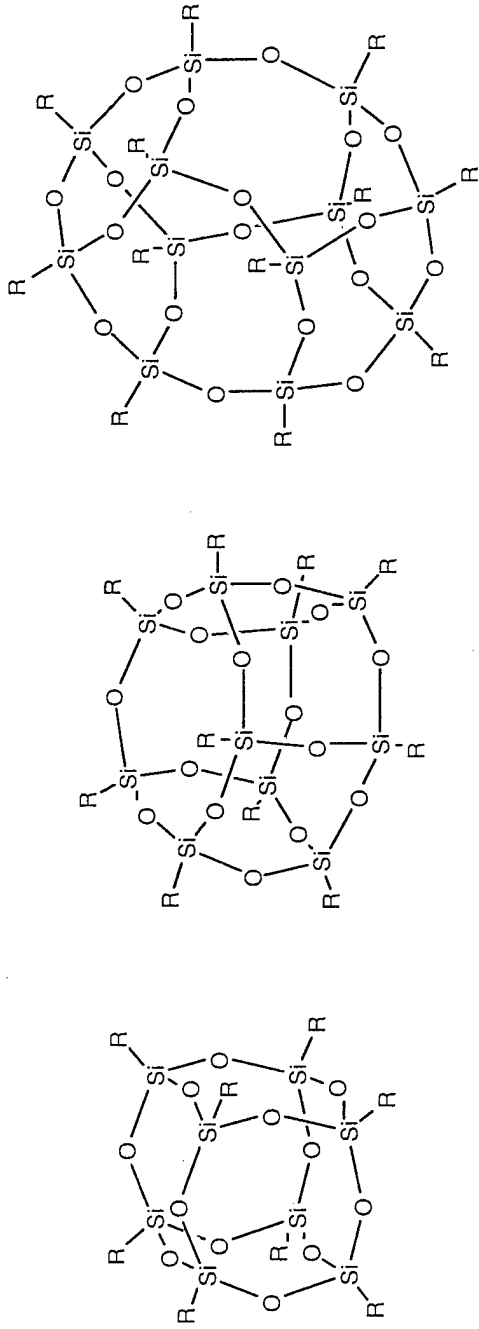


POSS-EPOXY

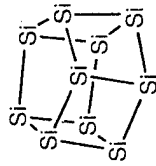


POSS-PMMA

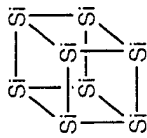
POSS Blended into Plastics



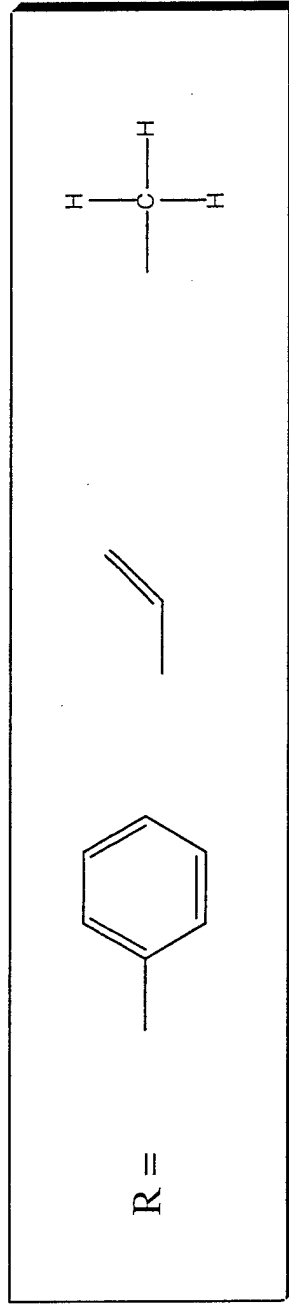
T₁₂



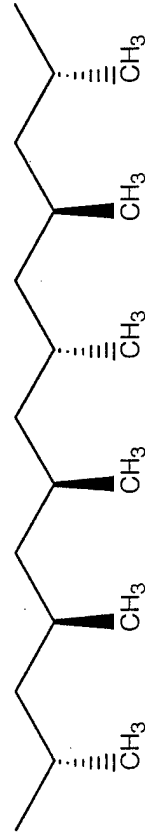
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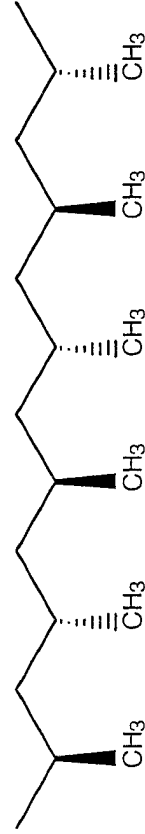
T₈



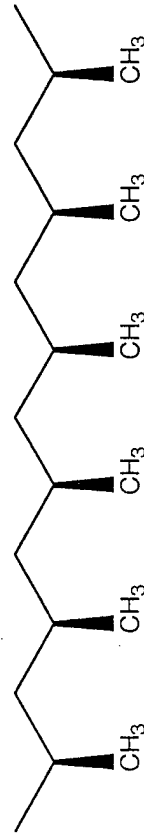
Materials Selection: Polypropylene and POSS



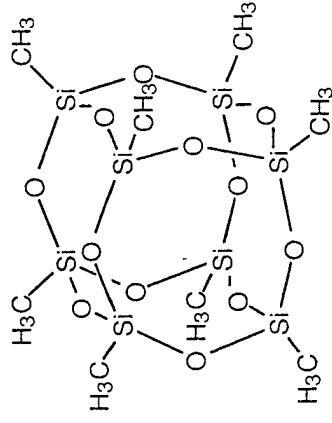
atactic polypropylene



syndiotactic polypropylene



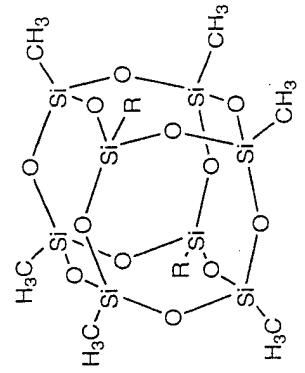
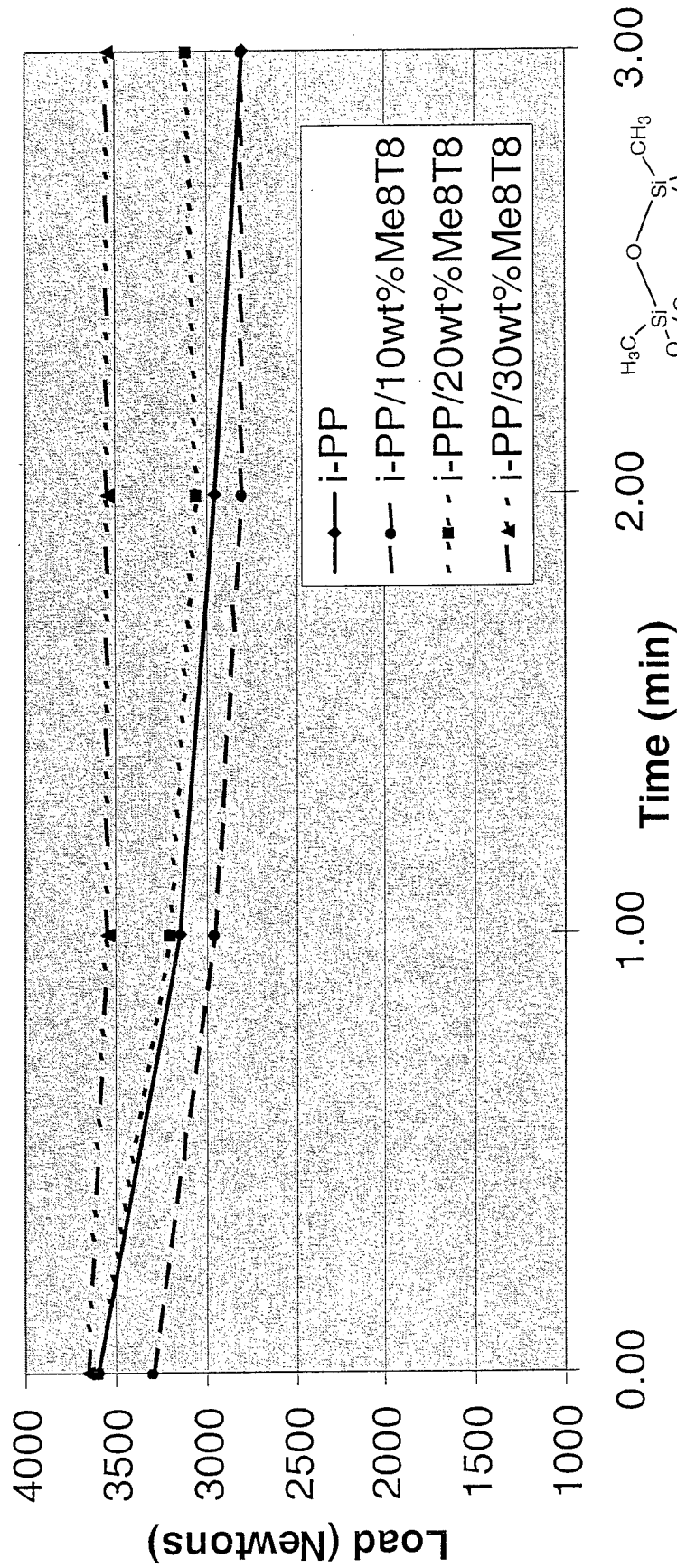
isotactic Polypropylene



Methyl₈T₈

i-PP/Me₈T₈ Processing Studies

iso-Polypropylene w/ Me8T8

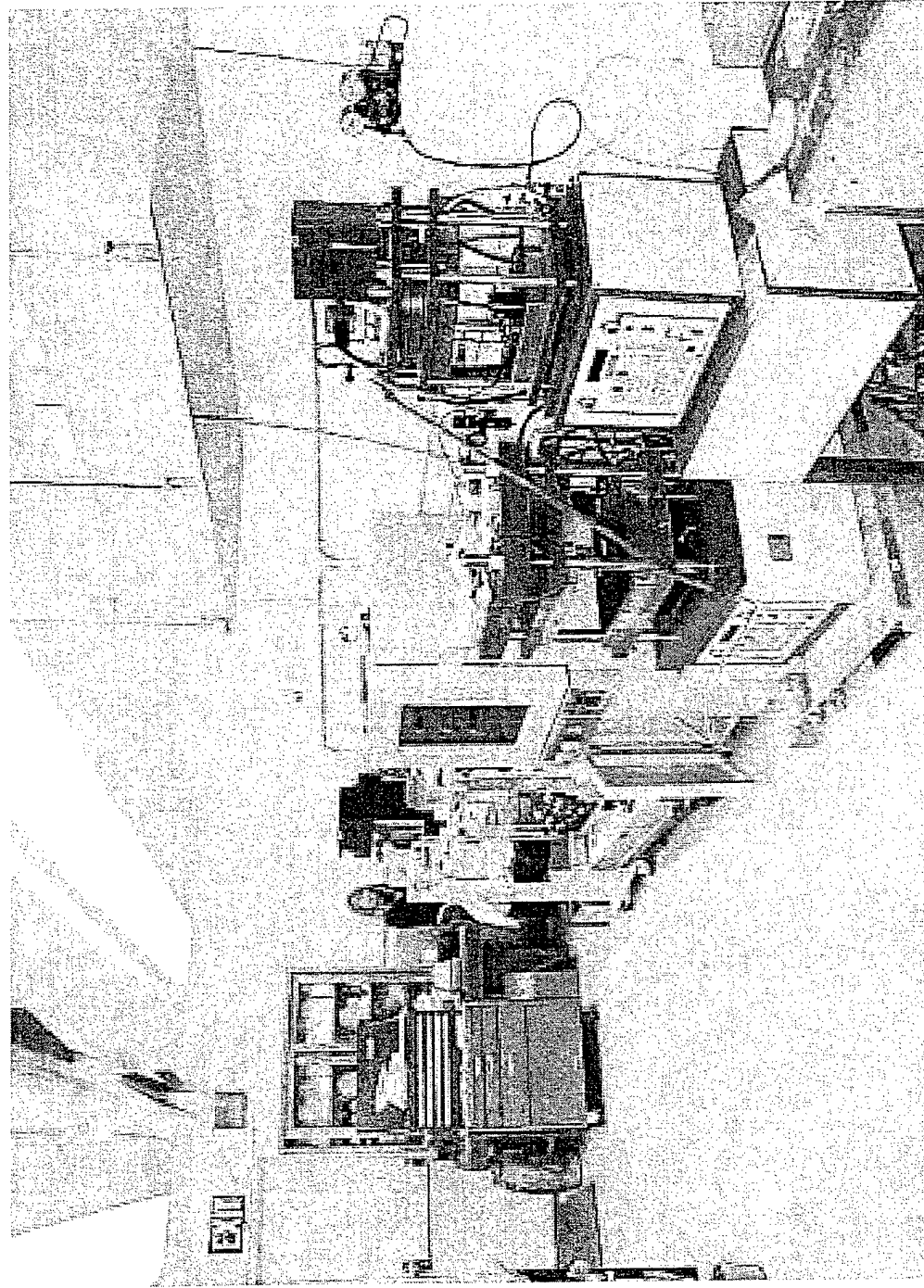


Prof. Andre Lee - Michigan State University

	Dow data	Neat <i>i</i> -PP (processed)	<i>i</i> -PP blended 2 wt% Methyl ₈ T ₈	<i>i</i> -PP blended 5 wt% Methyl ₈ T ₈	<i>i</i> -PP blended 10 wt% Methyl ₈ T ₈
Tensile Strength @ Yield; ASTM D638	5000 psi (34.5 MPa)	4800 psi (33.0 MPa)	5000 psi (34.5 MPa)	5100 psi (35.1 MPa)	5200 psi (35.8 MPa)
Flexural Modulus (0.05 in/min, 1% secant); ASTM D790A	240,000 psi (1.655 GPa)	235,000 psi (1.620 GPa)	251,000 psi (1.730 GPa)	255,000 psi (1.757 GPa)	262,000 psi (1.80 GPa)
HDT @ 66 psi, as injected; ASTM D648	210 °F (99 °C)	210 °F (99 °C)	221 °F (105 °C)	239 °F (115 °C)	255 °F (124 °C)
Impact Izod @25C ASTM D256A	0.5 ft-lb/in	0.55 ft-lb/in	0.55 ft-lb/in	0.62 ft-lb/in	0.75 ft-lb/in

- 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

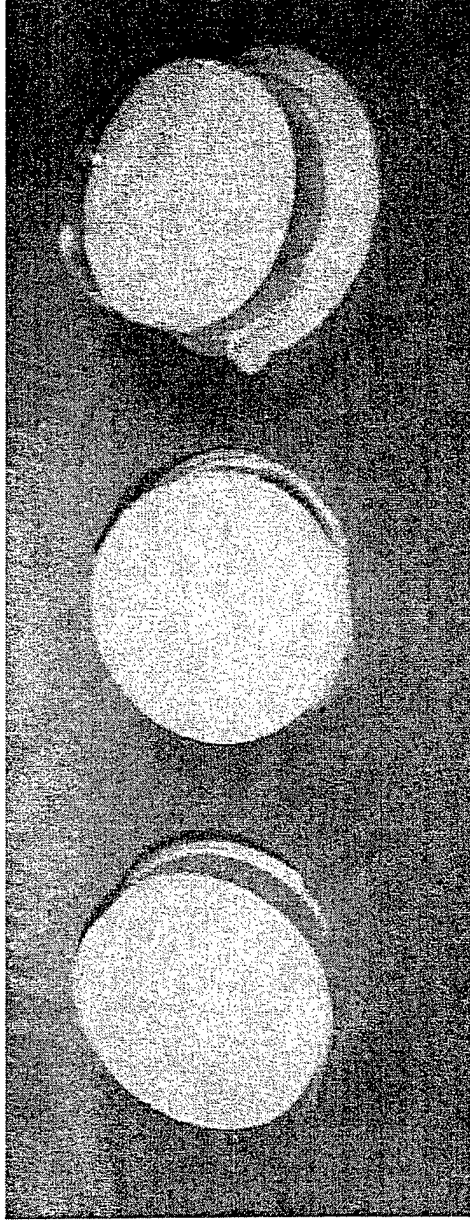


Polymer Processing Parameters

❖ Time

❖ Pressure

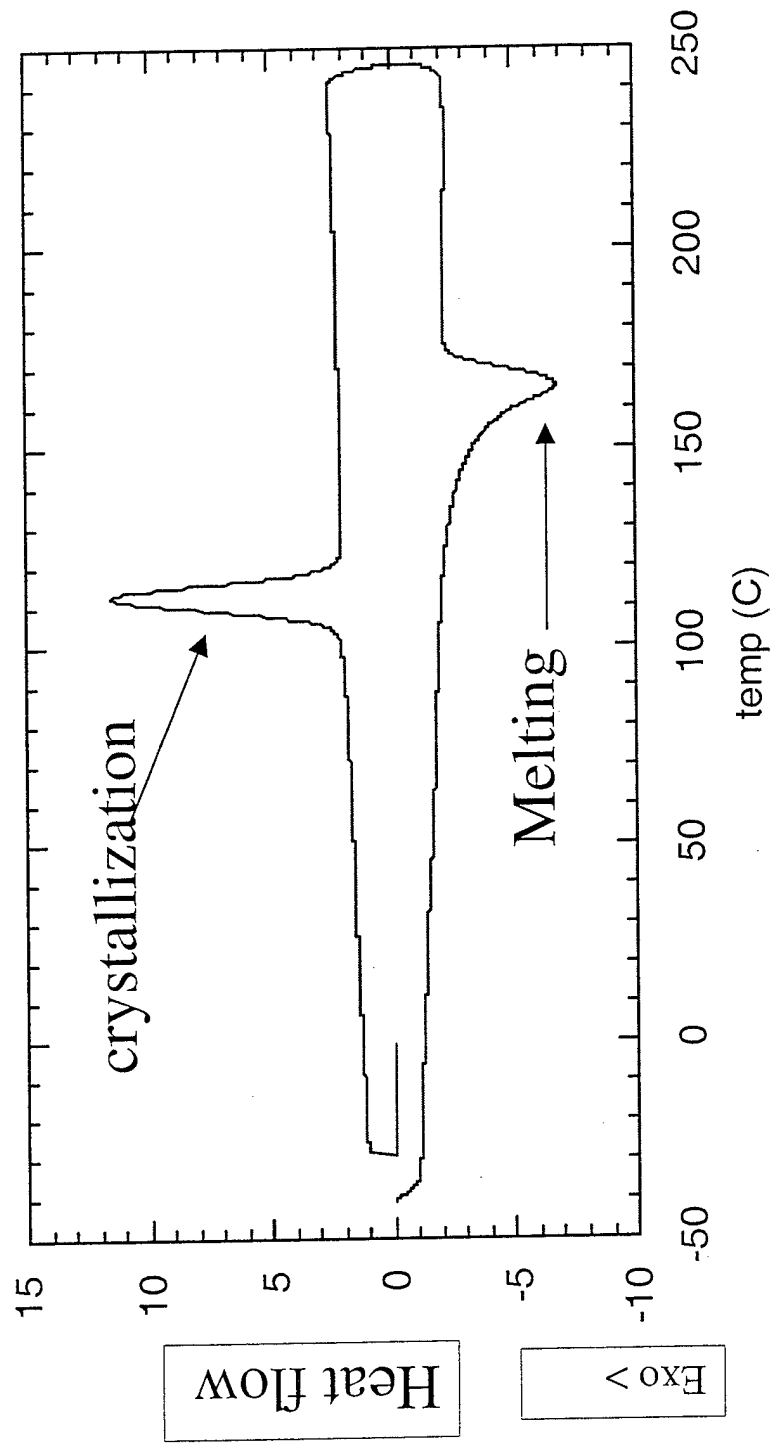
❖ Temperature



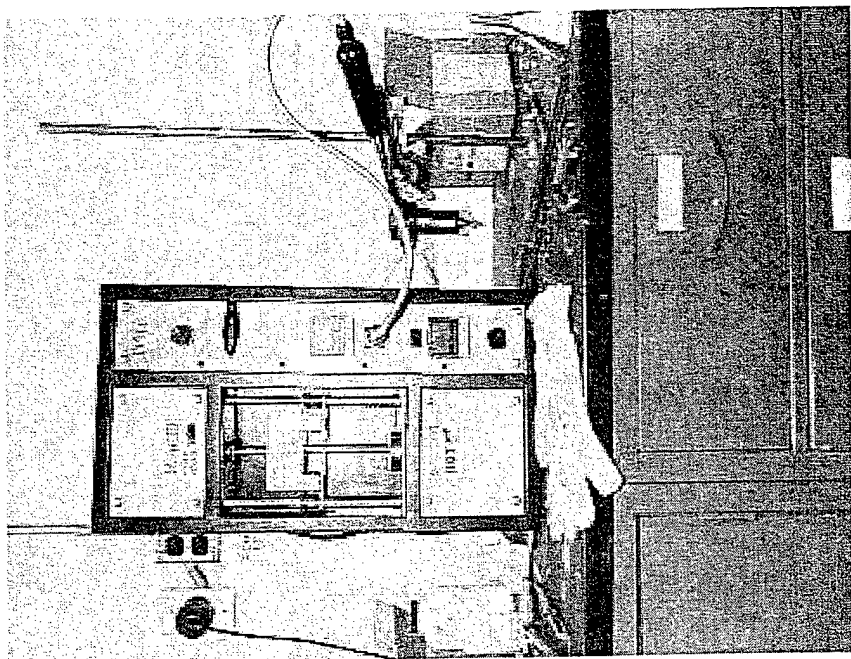
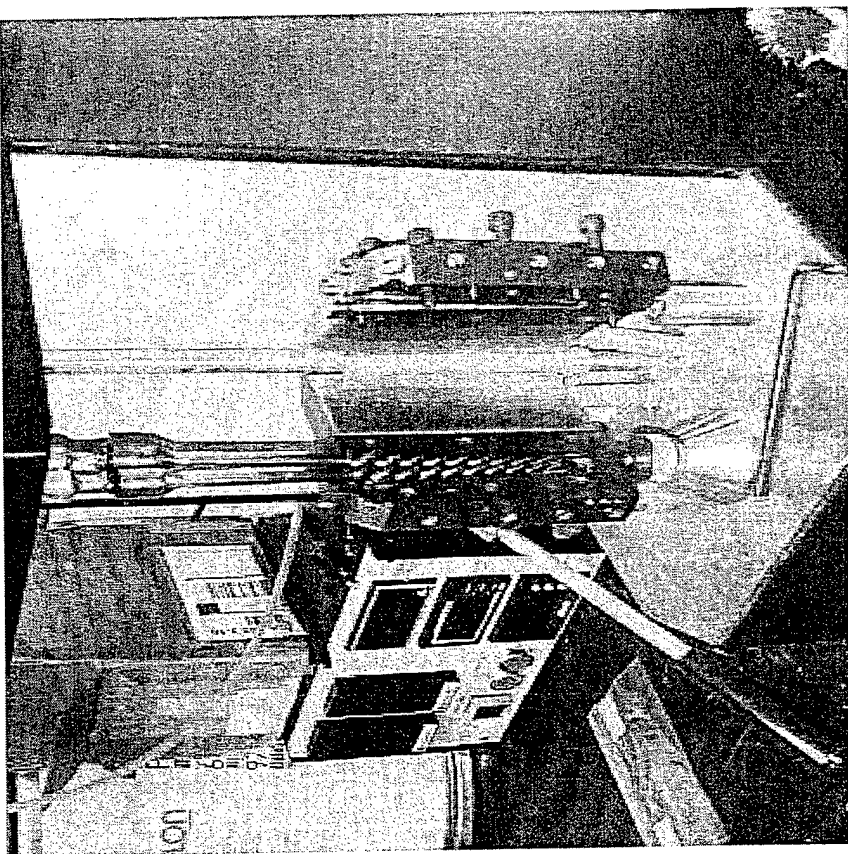
Procedure

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

Polypropylene DSC



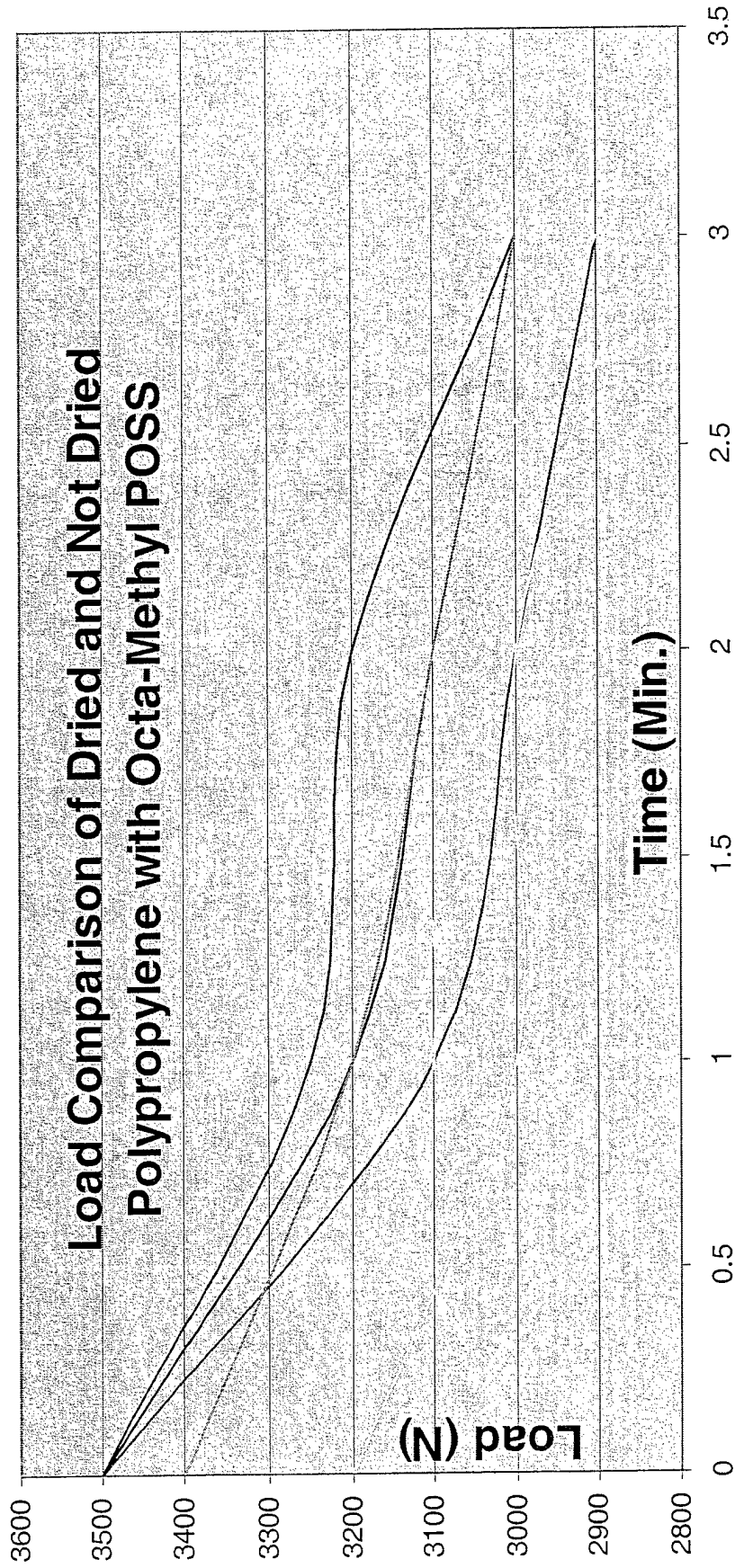
DACA



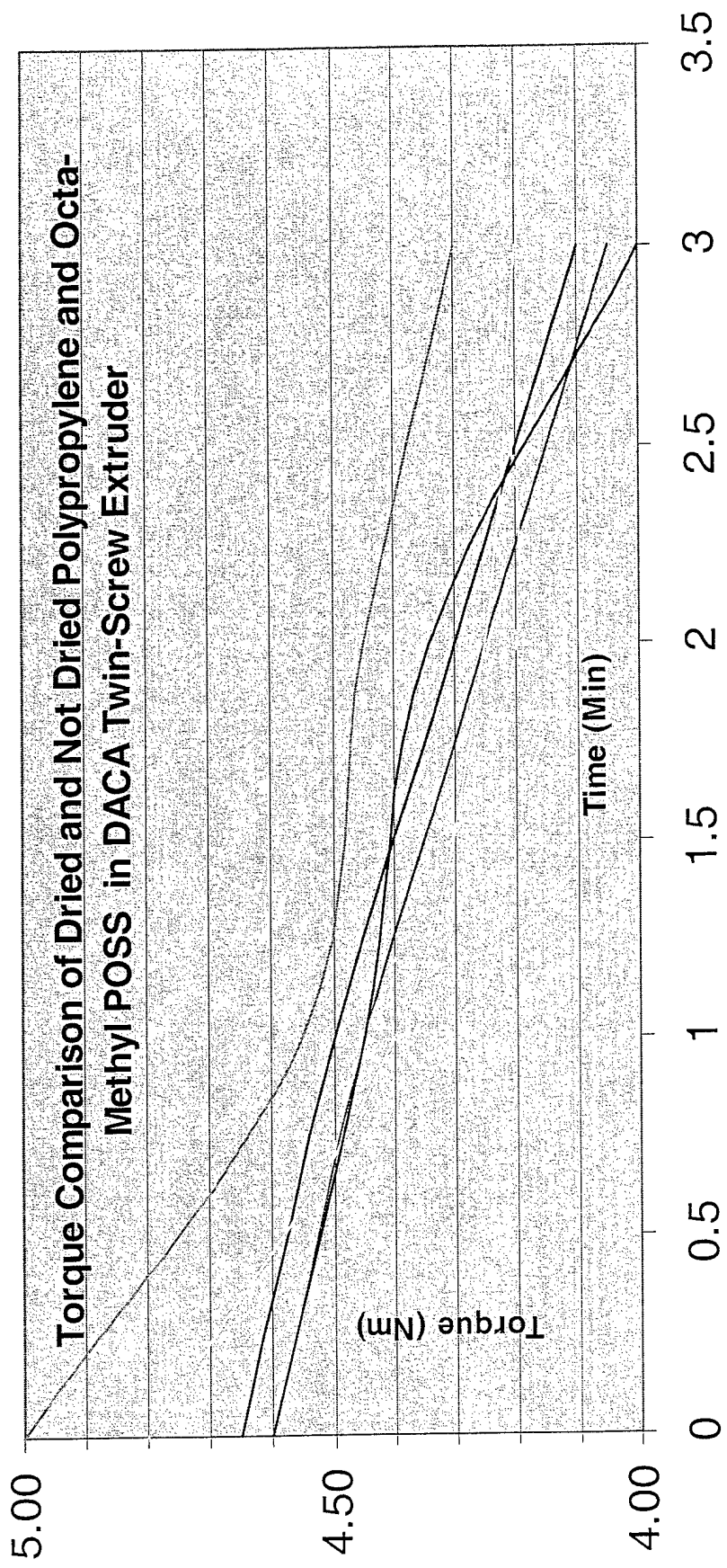
DACA Twin Screw Processing Parameters for Me8T8/iPP nanocomposite blends.

Mix #	Material Percentage		Load (N)			Torque (Nm)				
	PP	Me ₈ T ₈	Mix Duration (min)			Mix Duration (Min)				
1	Not Dried	Not Dried	0	1	2	3	0	1	2	3
2	100		3500	3200	3100	3000	4.65	4.50	4.30	4.10
3	100		3500	3100	3000	2900	4.60	4.45	4.25	4.05
4	90	10	3200	3000	3000	2850	4.80	4.40	4.25	4.20
5	90	10	3200	3100	3100	2900	4.60	4.45	4.20	4.25
6	90	10	3500	3250	3200	3000	5.00	4.55	4.45	4.30
	90	10	3400	3200	3100	3000	4.60	4.45	4.34	4.00

Hot Press

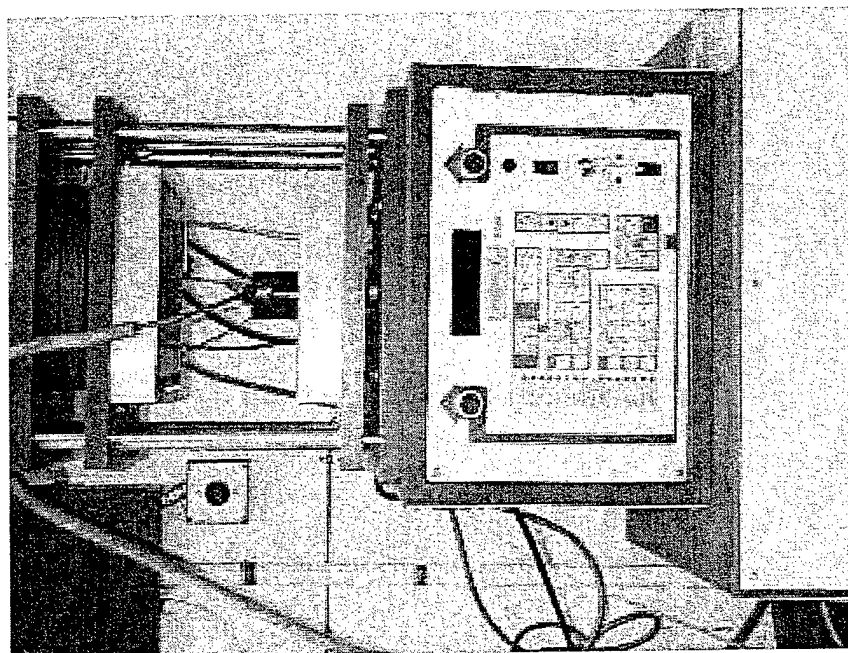


Dried Polypropylene	Not Dried Polypropylene
Dried PP, Dried POSS	Not Dried PP, Dried POSS
Dried PP, Not Dried POSS	Not Dried PP, Not Dried POSS



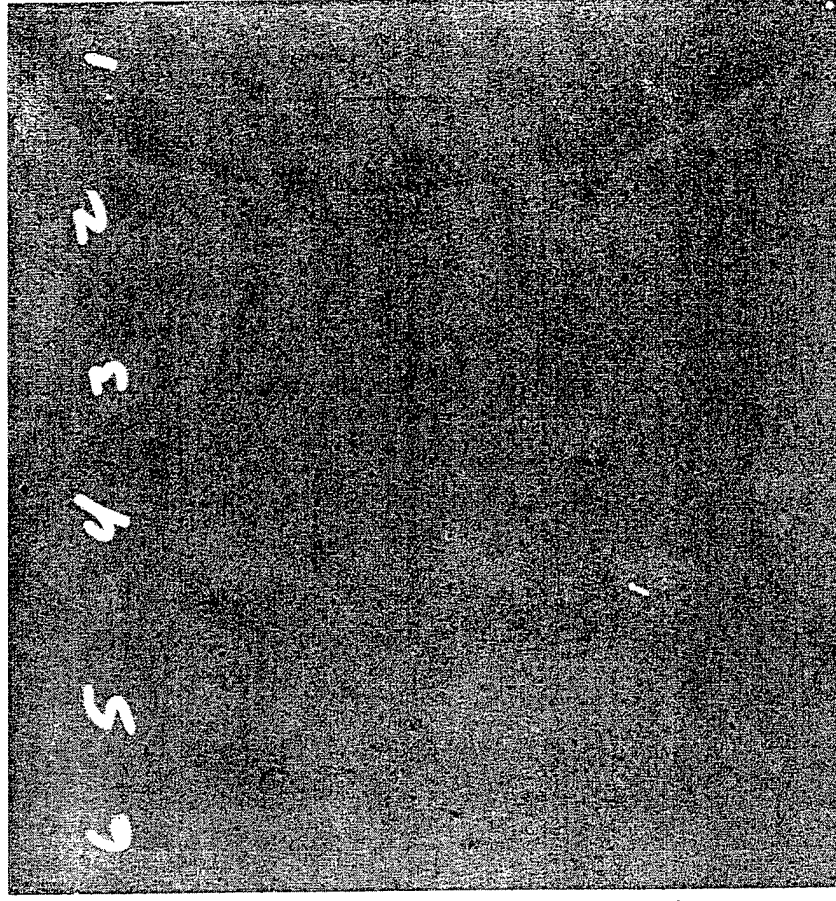
Dried Polypropylene	Not Dried Polypropylene
Dried PP, Dried POSS	Not Dried PP, Dried POSS
Dried PP, Not Dried POSS	Not Dried PP, Not Dried POSS

One Ton Press



Pressed film of DACA extruded POSS/PP blend variants

- 1 Dried PP
- 2 Not 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



10% POSS

0% POSS

SUMMARY

Drying seems to play a roll in making Me_8T_8 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 number 3 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.**