

**Increased Productivity** with Natural Additives

Vitrolite<sup>®</sup>



### VITROLITE®: A Novel Polymer Processing Technology

Steve Houlston

## VitroCo, Inc.

#### Company

- Hi Tech Environmental Products, LLC founded in 1997
- VitroCo, Inc. formed as subsidiary of VitroTech Corp. February 2004
- Headquartered in Santa Ana, CA

#### Materials technology emphasis

– How can we make existing chemical processes better with natural materials?

#### Global sales and distribution

- United States
- Canada
- Mexico
- UK, Benelux, Germany
- Italy
- Australia, Japan





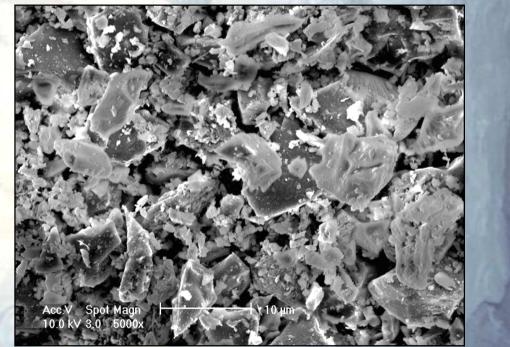
### What is Vitrolite<sup>®</sup>?

 Vitrolite<sup>®</sup> made by proprietary process from volcanic ash

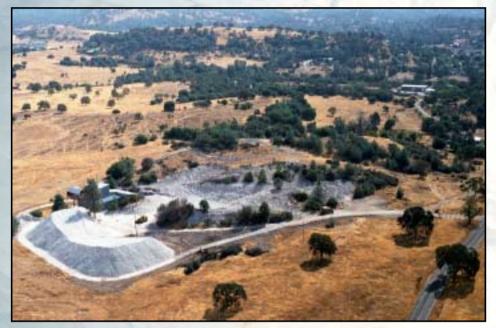
Key physical characteristics:

- Median particle size =  $7.5\mu m$
- White to off-white color
- Glass content >87-95%
- Hardness: 5-5.5 (<TiO<sub>2</sub>)
- Specific gravity: 2.3-2.4
- Index of refraction: 1.495
- Inflammable, nontoxic
- Low oil absorption (37-42 per ASTM D281)





### Vitrolite<sup>®</sup> Ore Reserves



 Adequate reserves for 80 years, at plastics market penetration of 20% Identified reserves

 in 3 mines of > 35
 billion pounds



### How is Vitrolite® used?

#### Typical usage

For most polymer formulations, a concentration of 0.5 weight percent is recommended

#### Typical application methods

- Directly compounded into polymer with other additives
  - Introduce along with other additive(s) of low concentration during compounding
  - Most effective because of superior dispersion and distribution
- In masterbatch such as with pigment
- Dry blend of concentrate pellets (weigh feeder is optimal)



### What does Vitrolite® Do?

### "Reduce Cost and Increase Productivity"

Part	Grams	Productivity gain	Cost w/o Vitrolite®	Cost with Vitrolite <sup>®</sup>	Cost savings/part
55 Gallon Drum	10260 gr.	16.46%	\$17.50	\$16.18	(\$1.32)
Television Case	7500 gr.	26.32%	\$22.72	\$22.00	(\$0.72)
Speaker Grill	227 gr.	36.17%	\$3.17	\$2.47	(\$0.69)
TV Cabinet Stand	et Stand 757 gr. 47.46%		\$3.03	\$2.61	(\$0.41)
Door Panel	908 gr.	24.24%	\$2.00	\$1.86	(\$0.14)
Battery Case	741 gr.	25.90%	\$1.62	\$1.48	(\$0.13)
Seal Support Ring	8 gr.	55.05%	\$0.25	\$0.16	(\$0.08)
Visor	467 gr.	7 gr. 25.65% \$1.08 \$1.02		\$1.02	(\$0.06)
Visor	463 gr.	30.00%	\$0.88	\$0.86	(\$0.02)



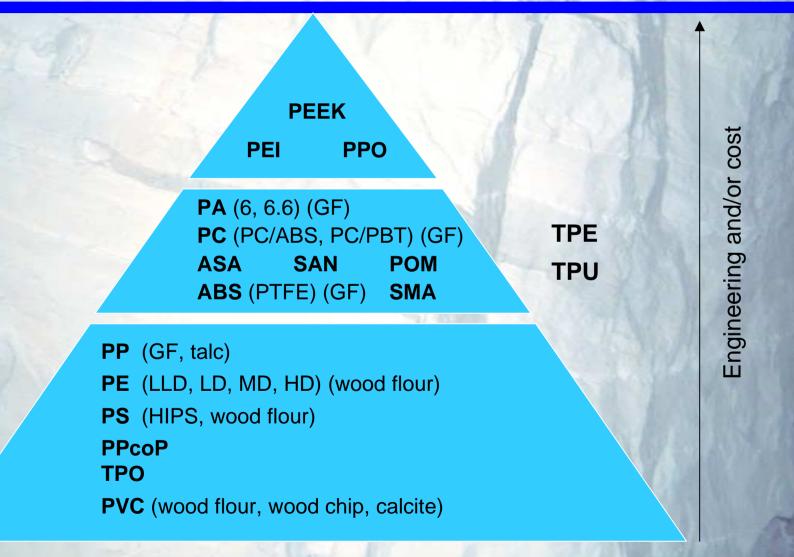
# Why is this processing technology NOVEL?

A low concentration of micron-scale, non-toxic aluminosilicate glass particles alter polymeric liquid viscosity without adversely affecting polymer composition or additives

The altered viscosity provides multiple benefits for any polymer composition or processing method

The novel processing technology is subject to two pending patents.

## 5 Effective in all polymers tested



### **Effective in All Processes**

- Injection molding
- Extrusion molding
- Blow molding
- Compression
- Rotomolding
- Thermoforming
- Slush molding
- Blown Film



#### AMORPHOUS

RESIN	PART	START TEMP °C	END TEMP °C	DIFF °C	% PROD INCREASE
ABS	AUTO	241	238	-3	16
ABS	PILLAR	264	259	-5	20
ABS	CONSOLE	238	232	-6	39
ABS	CONSOLE	238	232	-6	50
PC	AUTO	-	SAR		15
PC	COVER	213	194	-14	43
РОМ	GEAR	204	189	-15	16
ASA	GRILLE	204	189	-15	22
ASA	ASA BOX		210	-31	22
PETG	САР	2 <mark>66</mark>	253	-13	31
PEI	FIXTURE	359	359	0	20



Automotive Components



#### **AMORPHOUS—ALLOYS & FILLED**

RESIN	PART	START TEMP °C END TEMP °C DIFFERENCE °C %		% PROD INCREASE	
PC/ABS	HEADER	282	260	-22	15
PC/ABS	PANEL	278	273	-5	26
PC/ABS	GRILLE	260	252	-8	33
PC/PBT	BUMPER	248 242		-6	58

PC25%GL	FRAME	325	315	-10	23
ABS-GL	SUPPORT	239	227	-12	27
ABS10%PTFE	HANDLE	232	224	-8	32
SMA15%GL	PANEL			- 23	16



Pails and Containers



#### **SEMI-CRYSTALLINE**

RESIN	PART	START TEMP °C	START TEMP °C END TEMP °C DIFFERENCE °C		% PROD INCREASE
PP	VISOR	229	219	-10	16
PP	САР	208	203	-5	21
PP	SEAT	203	198	-5	26
PP	HANDLE	210	193	-17	28
PP	VISOR	229	219	-10	30
PP	PANEL	212	212	0	32
HDPE	PAIL	310	240	-70	29
HDPE	PALLET	230	215	-15	29
HDPE	PAIL	244	201	-43	17
HDPE	PAIL	253	226 <b>-27</b>		13
PA	FITTING	308	284	-24	25
PA6/6	ELEC. TIE	-6			23



#### **SEMI-CRYSTALLINE—IMPACT MODIFIED & FILLED**

RESIN	PART	PART START TEMP °C E		END TEMP °C DIFFERENCE °C	
PPcoP	PANEL	201	198	-3	16
PPcoP	CONSOLE	207	199	-8	19
PPcoP	САР	236	236	0	23
PP-NR	COVER	241	241	0	21
PP-NR	DOOR	231	231 0		24
PP-NR	COVER	241	226	-15	36
	N. N.	12.00	100		0.03/10/50
PP-TALC	DUCT	194	194	0	15
PP25%TALC	TALC VISOR 228		221	-7	25
PP40%TALC	HOUSING	239	227	-12	24
PA33%GL	FAN	254	254	0	16



#### **SEMI-CRYSTALLINE AND AMORPHOUS**

RESIN	PART	START TEMP °C	END TEMP °C	DIFFERENCE °C	% PROD INCREASE
HDPE	SHEET	343 · · · ·	1.00		27
		A CONTRACTOR OF THE OWNER	And ha	144	A DI AND A DIA
PS	LINER	188	178	-10	14
HIPS	SHEET	165	157	-8	11
PVC	TUBING	152	147	-5	25
PVC	TUBING	152	147	-5	54
		1971-00		1033010	
PVC (FR)	CABLE	172	172	0	64
PVC (FR)	CABLE	168	157	-9	100
PVC (FILLED)	PIPE	170	170	0	20
PVC (FILLED)	PIPE	162	155	-7	58
PVC (FILLED)	PIPE	162	155	-7	66
PVC-WOOD	LUMBER	14 - A - A - A - A - A - A - A - A - A -			75





### **BLOW & THERMOFORMING**

RESIN	PART	START TEMP °C END TEMP °C		DIFFERENCE °C	% PROD INCREASE
HDPE	JUG	179	179	0	13
HDPE	BOTTLE	238	238 229 <b>-9</b>		14
HDPE	DRUM	193	183	-10	15

	1		1.70	A REAL PAR	1.038/10.2507
HIPS	REF. LINER	free to a	7 11	the second second	23



#### USP Class VI Compliant

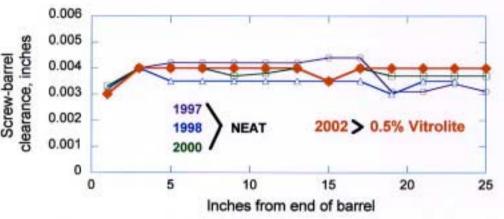
- USFDA Compliant
- Recognized as safe in all world markets



### Safe for Equipment

#### Does NOT cause abrasion

- No change in screw/barrel dimensions after 350,000 parts
- Abrasion unlikely:
  - Vitrolite<sup>®</sup> softer than hard chrome
  - Low particle concentration
  - Small particle diameter
  - Equant particle shape



### Safe for long-term and Automotive applications

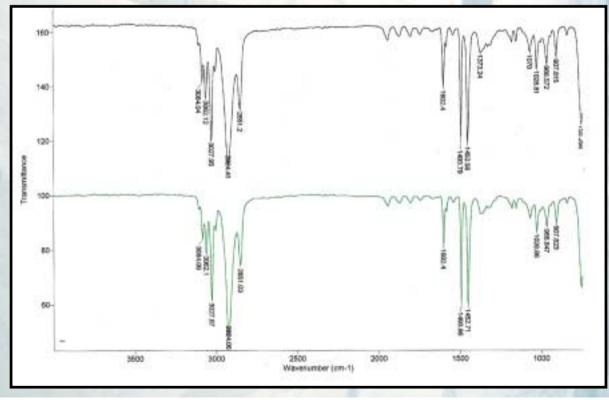
- Accelerated ageing
  - No change at 1000 hrs for HIPS
- Automotive testing
  - No changes in polymer performance during and after life-cycle tests
  - Includes accelerated ageing

AUTOMOTIVE	TESTS PASSED
Warm odor	Deformation-life cycle
Flammability	Loading-life cycle
Fogging (vinyl)	Heat ageing
Light stability	Dimensional stability-life
	cycle
Cleaning ability	Cold crack and impact
Vertical loading-life	Drop
cycle	

### Appropriate for Electronics Applications

- Underwriters Laboratories UL 746A Flammability Test--Passed
- Ash Content Test Passed with greater than 95% residual ash and no combustion
- FTIR (Fourier Transform Infrared Spectroscopy) Passed



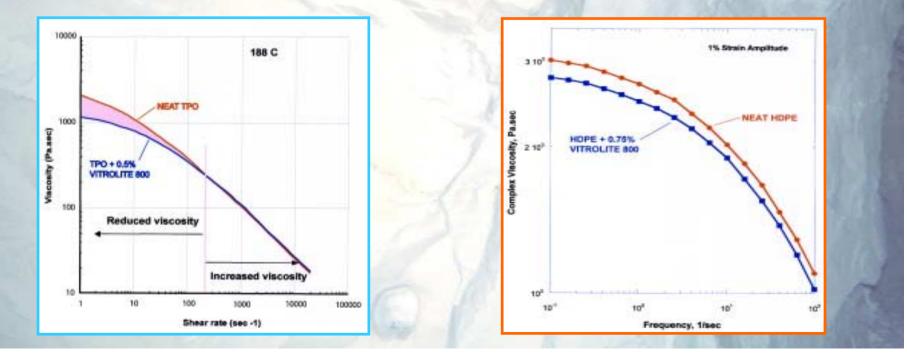


### The "Vitrolite<sup>®</sup> Effect"

The "Vitrolite® Effect" describes the <u>multiple</u> effects of Vitrolite® on polymeric liquids at stresses and shear-rates typical of finishing environments.

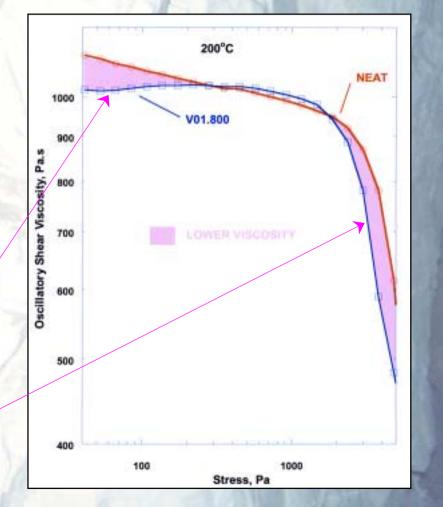
### Effect of Vitrolite<sup>®</sup>-steady state and dynamic

- Vitrolite<sup>®</sup> substantially *decreases* viscosity in low stress/low shear rate environment:
  - Steady-state capillary die measurements clearly show viscosity reduction at low shear rates for TPO
  - Low-strain amplitude (linear regime) measurements on oscillating plate rheometer show viscosity reduction for HDPE



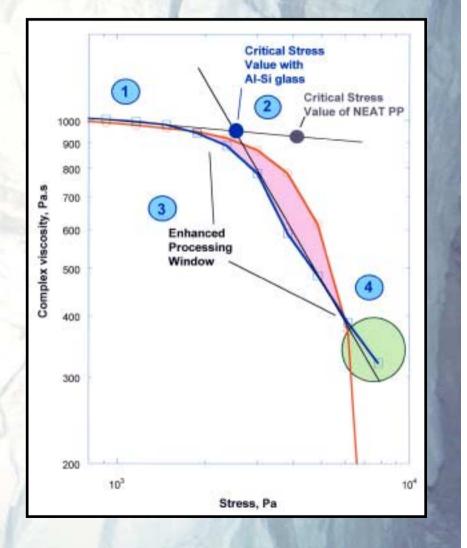
### Dynamic melt viscosity with Vitrolite<sup>®</sup>

- Melt viscosity from oscillatory shear
  - Use variable stress amplitude at fixed oscillation frequency
  - Allows for determination of viscosity in linear to non-linear regimes
- For NEAT PP
  - Addition of 0.5% Vitrolite<sup>®</sup> results in two regions of reduced viscosity
    - Low stress amplitudes (approximately linear regime)
    - High stress amplitudes (non-linear regime)



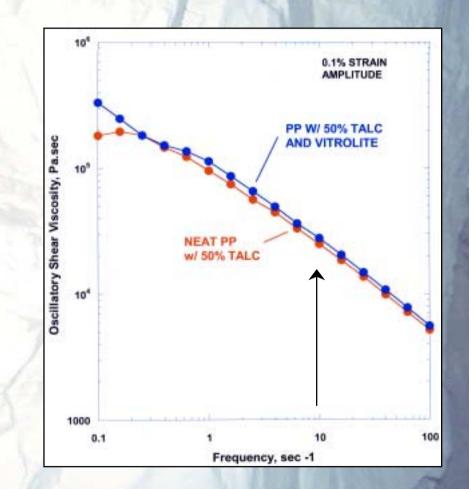
### The "Vitrolite® Effect" at high stress amplitude

- 1. No increase in viscosity
- 2. Decrease in critical stress amplitude
- 3. Enhanced Processing Window (EPW)
  - Viscosity of polymer with Vitrolite<sup>®</sup> is less than that of NEAT
- 4. Higher viscosity at very high stress amplitude



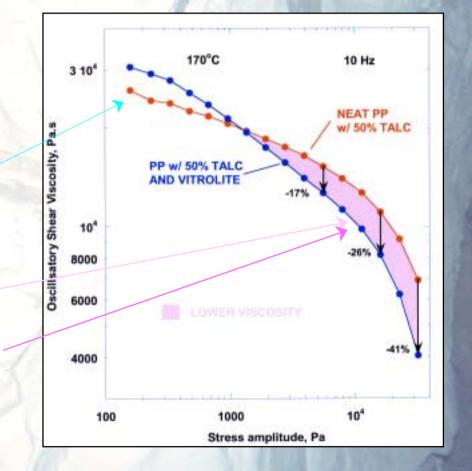
### Melt viscosity with Vitrolite<sup>®</sup>--filled polymers

- Effect of Vitrolite<sup>®</sup> in linear regime is as expected
  - Addition of more solid increases the melt viscosity slightly
- How does Vitrolite<sup>®</sup> affect non-linear viscosity?
  - Fixed frequency of 10 sec<sup>-1</sup> and vary stress amplitude



### Melt viscosity with Vitrolite<sup>®</sup>--filled polymers

- Melt viscosity of filled polymers are substantially affect by Vitrolite<sup>®</sup> in nonlinear regime
  - Viscosity is higher at low stress amplitudes of linear stress-strain rate regime
  - HOWEVER, polymer melt viscosity is substantially LOWER in non-linear regime



# Implications of reduced viscosity

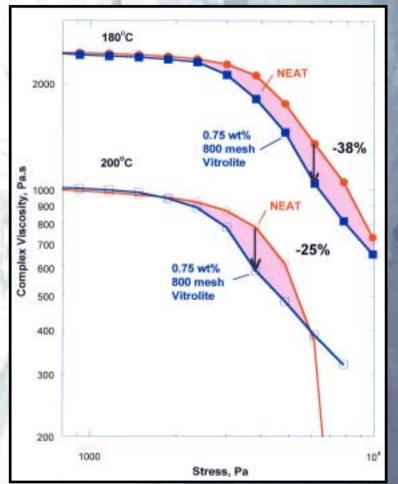
- Why is Vitrolite<sup>®</sup> effective in all polymer processing environments?
  - Vitrolite<sup>®</sup> results in lower melt viscosity extending across an extremely wide range of stresses and strain rates
    - Unanticipated result particularly at low strain rates
  - Vitrolite<sup>®</sup> can be effective in flow-restricting region of equipment, thus its broad applicability to all processing environments
- Many other advantages accrue from use of Vitrolite<sup>®</sup>



### Why take advantage of the "Vitrolite® Effect"?

# Processing temperature reduction

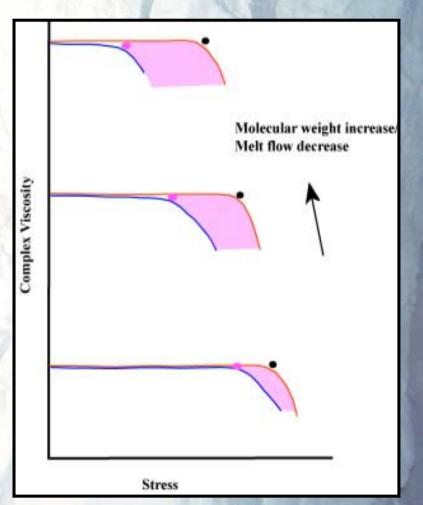
- Lowering the melt temperature has anticipated result of increasing NEAT polymer viscosity (red)
- Unanticipated result for polymer with Vitrolite<sup>®</sup> (blue):
  - Reduction in viscosity INCREASES at lower temperature and is apparent over wider stress range
- Implications
  - Polymers can be processed at lower temperature with little penalty of higher viscosity
  - Less polymer and additive degradation
  - Higher production rate



### Allows use of polymers with higher molecular weight

Another unanticipated result is that the "Vitrolite" Effect" increases with increasing molecular weight

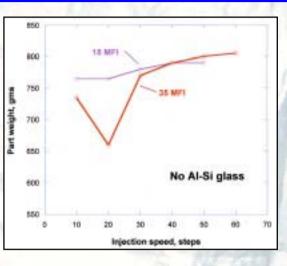
- Lower critical stress value
- Larger stress range for EPW
- Larger viscosity decrement
- First Implication
  - Lower melt polymer with Vitrolite<sup>®</sup> can be processed as readily as higher melt NEAT polymer

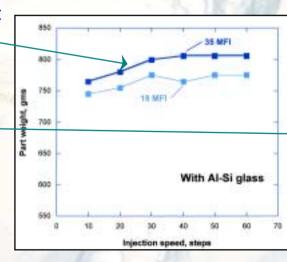


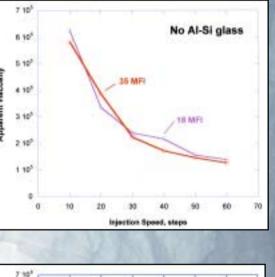
## Reduces effect of molecular weight on processing

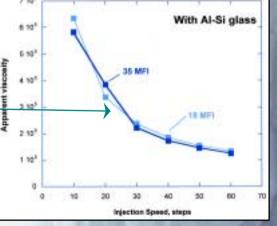


- Stable molding process not affected by "wide-spec" polymer or lot-to-lot variation
  - Part weight consistent over wider injection \_\_\_\_\_\_
     speed range
  - Apparent viscosity differences are negated







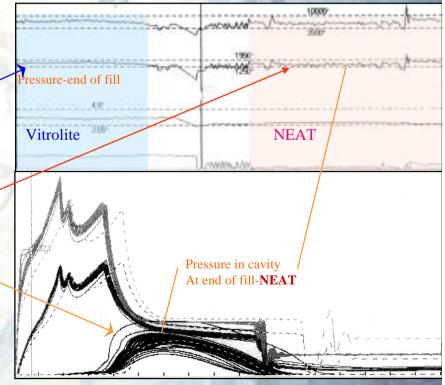




### **Better Parts--injection**

- Dimensional accuracy and precision critical for many applications
  - Accuracy = mold dimensions
  - Precision = repeatability of molding process
- Vitrolite<sup>®</sup> greatly improves precision
  - Weight variation of closures greatly reduced
- Improved precision traceable to improved mold fill
  - Pressure in cavity varies far less with Vitrolite (left) than without (right)
  - Without Vitrolite<sup>®</sup>, cavity plastic pressure varies substantially
    - VARIABLE PART DIMENSIONS
    - LONG TERM DIMENSIONAL INSTABILITY

POLYMER	Grams	%	Change	
		+/-	in % +/-	
PP	18.595	0.29		1.5
w/ Vitrolite	18.624	0.15	- 0.14	10
<b>PP + 40% talc</b>	24.243	0.39		1
w/ Vitrolite	24.364	0.23	-0.16	1.0
PC	25.270	0.03		100
w/ Vitrolite	25.270	0.03	0.00	100
PC/ABS	24.683	0.30		27
w/ Vitrolite	24.674	0.04	- 0.26	32
PA66 + 33%	28.047	0.31		19.1
glass				120
w/ Vitrolite	27.970	0.20	- 0.11	120
		100 B		and the second





### **Better Parts--extrusion**

- Profile extrusion dimensions are more precise
- Wire and cable jackets with high concentration of non-halogenated fire retardant are more consistent
  - Variation in 0.0625 mm jacket reduced from +/- 0.012 to +/- 0.004 (-67%)
  - Ovality index reduced from 0.00019 to 0.00005 (-75%)
  - Failure rate for individual batches reduced to zero

### Polymer properties are marginally affected

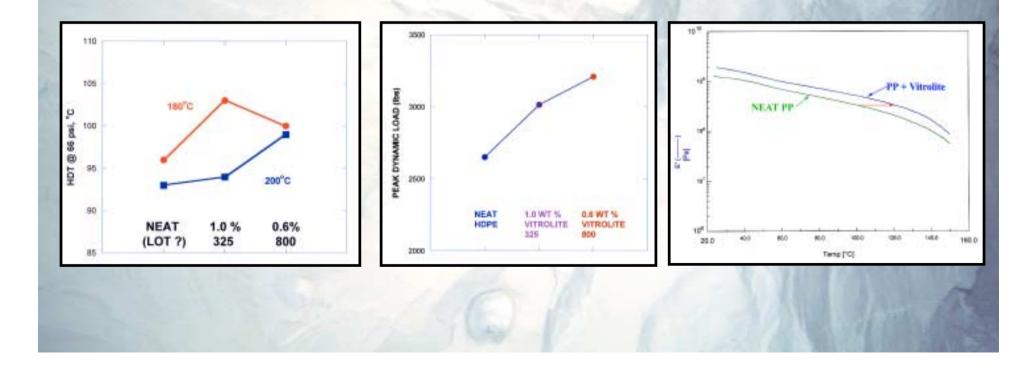
Physical/mechanical properties of polymer processed with Vitrolite<sup>®</sup> are not changed when processed at *typical* melt temperature

	Polymer		P	P	AI	BS	PC/A	ABS
Property	Units	Reference	NEAT	0.5% Vitro	NEAT	0.5% Vitro	NEAT	0.5% Vitro
Melt Flow	gm/10 min	ASTM D1238	29.6	29.4	5.9	7.2	6.9	7.1
Impact Dyna	J, 23°C	ASTM D3763.02	31.2	36.4	30.3	29.3	56.7	60.5
tup	-30°C		5.7	5.4	17.5	19.7	68.9	62.9
Impact Notched	J/m, 23°C	ASTM D238	56.4	57.4	163	158	591	637
Izod	-30°C		28.5	3.6(?)	77	74	456	380
Impact	kJ/m, 23°C	ISO 179	4.1	4.2	8.4	8.9	52.8	51.0
Charpay	-30°C		4.8	3.5	5.1	5.8	18.9	37.3
HDT	°C, 0.45 Mpa	ASTM D648	81.9	84.5	90.0	90.0	126.7	126.3
	1.82 Mpa		53.5	52.5	78.1	78.2	101.0	100.6
Tensile @ yield	Мра	ASTM D638	24.8	24.3	36.6	36.4	46.1	47.2
Flex modulus	Мра	ASTM 790	1364	1350	2180	2176	2124	2097

Similar results for HDPE, PA, PC and PEI

## Polymer properties may be improved

Physical/mechanical properties of finished polymer such as impact strength, heat deflection temperature (HDT), flexural modulus and elastic modulus (E') may be improved at a *lower* processing temperature made possible by Vitrolite<sup>®</sup>



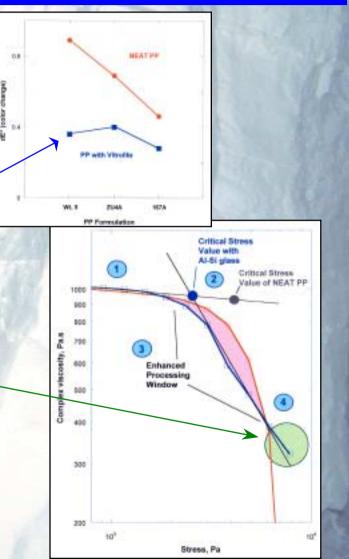
# Improved additive dispersion

#### Additive performance may be improved

- Olefin- and styrene-based nonhalogenated fire-retardant compounds require 20-30% less additive with Vitrolite<sup>®</sup> to achieve same FR rating
- Comparable results with other molecular and solid additives (e.g., UV stabilizers and pigments)

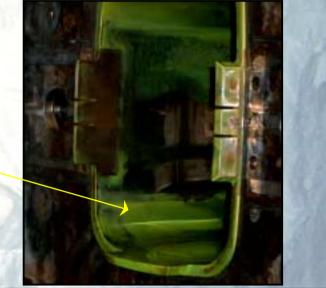
#### ♦ Why?

- Vitrolite<sup>®</sup> does not have same function as additive
- Relatively higher viscosity in highstress/high-shear rate mixing improves dispersion and distribution of additives, thus making them perform better
- Implication
  - Additive concentration may be reduced without affecting performance (may lower costs)



## Reduced contamination of processing equipment

- Pigments, additives and polymer degradation products can coat wetted surfaces
  - In one application, yellow pigment coated mold surfaces
  - How do you prevent and/or correct?
- Prevention effected by improved dispersion and mild purging action of Vitrolite<sup>®</sup> at typical (0.5%) concentration
- Correction effected by purge product of 50:50 Vitrolite<sup>®</sup>: carrier polymer
  - Only 1 lb for 610 ton press!







## How does Vitrolite<sup>®</sup> Interact with polymers ?

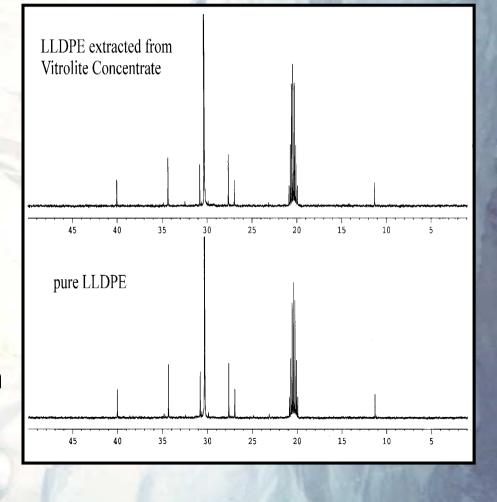
## Vitrolite<sup>®</sup> does not affect polymer integrity

- Analyze molecular weight distribution of polymer processed without and with Vitrolite<sup>®</sup> by High Pressure Liquid Chromatography/GPC
- RESULTS: NO detectable change in M<sub>w</sub>/M<sub>n</sub> of polycarbonate

Polymer	Extrude?	Weight % Vitrolite <sup>®</sup>	M <sub>w</sub> x 10 <sup>-4</sup>	M <sub>n</sub> x 10 <sup>-4</sup>	M <sub>w</sub> /M <sub>n</sub> 2.02	
PC	N		4.64	2.30		
PC Y			4.53	2.28	1.98	
PC	Y	1	4.56	2.28	2.01 1.99 2.01	
PC	Y	2	4.45	2.23		
PC	Y	5	4.42	2.21		

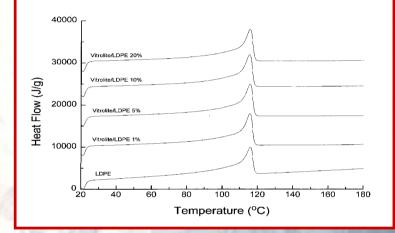
## Vitrolite<sup>®</sup> does not affect polymer integrity

- Chain scission or degradation byproducts, *if* created by Vitrolite<sup>®</sup>, should yield different NMR spectra
- RESULTS: High resolution <sup>1</sup>H and <sup>13</sup>C NMR spectra of NEAT LLDPE and extract from 50:50 concentrate are indistinguishable

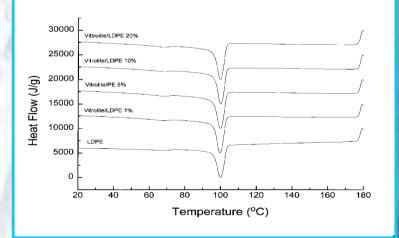


## Vitrolite<sup>®</sup> does not affect polymer integrity

 Crystallite nucleation or crystallization kinetics in semicrystalline polymers, if affected by Vitrolite<sup>®</sup>, should have different T<sub>m</sub> and T<sub>c</sub> and heat flow values during heating (upper) and cooling (lower) cycles



- RESULTS: heating and cooling curves are virtually identical other than anticipated decrease in heat flow
  - Comparable results for PP and nylon containing 5 to 10 times typical Vitrolite<sup>®</sup> concentration



## Vitrolite<sup>®</sup>-polymer interaction—*What is it?*

#### Not a chemical interaction:

- No dependence on polymer composition
- No demonstrable change to polymer integrity
- No known interference or reaction with additives
- Maintains full effects after multiple processing steps or recycling
- Primarily a physical interaction:
  - Viscosity alteration dependent on physical attributes of particles (size, shape, morphology, etc.)
  - On-going research into detailed mechanisms focusing on physical changes to molecular entanglement and/or changes to stress distribution in a heterogeneous medium
  - There appears to be an increase in the molecular spacing with the addition of Vitrolite.



### What Doesn't Work

Standard tests that will not show the effect:

- Standard-load melt indexing
- Capillary die rheometry with standard L/D ~ 20 dies
- Zero-length extensional viscometry
- Spiral Flow





### Measuring the "Vitrolite® Effect"

Dynamic-mechanical analyzers such as parallel-plate, oscillating rheometers

Variable analytical conditions:

- Frequency
- Stress amplitude
- ♦ Temperature

Results can be confirmed by steadystate high-load melt indexing and capillary die with L/D >33



## The "Vitrolite® Effect"

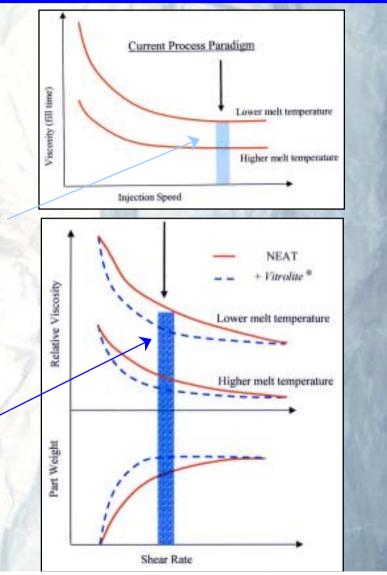
### How do you take advantage of the "Vitrolite® Effect"?

## "Seeing" the Vitrolite® Effect

- ONLY when molding conditions are optimal, the effect of Vitrolite<sup>®</sup> may be apparent:
  - Mold fill or flash -
  - Decrease in barrel pressure
  - Decrease in fill time/increase in extruder output
- Typically, however, process must be adjusted to take advantage of Vitrolite<sup>®</sup>

## Optimizing with Vitrolite<sup>®</sup>--Injection Molding

- Optimization requires specific procedure to change process parameters
  - Reduce heat in system to shorten cooling time
- Current Paradigm of injecting fast with lower melt temperature will not accomplish goal
  - Higher melt viscosity
- However, with Vitrolite<sup>®</sup>, a New Paradigm
  - Melt viscosity increase is mitigated by Vitrolite<sup>®</sup> IF shear rate is optimized for Vitrolite<sup>®</sup> Effect



## Advantages of the Vitrolite<sup>®</sup> Technology—Summary

- Effective in all polymer processing environments
- Effective in all unfilled and filled polymer compositions
- Reduced melt viscosity improves productivity and part quality while reducing rejects
- Non-reactive and safe in all applications
- Physical/mechanical properties of finished polymers are the same or improved
- Mitigates viscosity effects of higher molecular weight grades and molecular weight variation between lots
- Improves additive performance
- Prevents or corrects processing equipment contamination
- Likely reduction in electrical power usage per weight of polymer processed

## **Documenting the Process**

- Customer Information
  - Identifying Customer Needs
- Pre-Trial Audit
  - Meeting Customer Needs
- Demonstration Trial
  - Parameter Setup Form
  - Trial Data for Sales
  - Estimated Total Cost Benefit
- Production Audit
- Production Trial
- Production "Total Cost Benefit"
- Vitrolite Optimization Plan

Trial Date:				Contact:						
Company:				Trial Go						
Process:		Р	Press:			Tonnage:		Tool I.D. No./Cavities:		
Material: Pigment:		Product:						Trial Run Time:		
waterial.	i ignorit.		100	iuci.				Start	End	
								otart	Liiu	
Cost Elements		A c t	A v g	Costs	Baseline Cost/ Cycle		Vitrolite Cost/ Cycle	Percent Change		
MACHINE COSTS/ HOUR						\$0.00		\$0.00	0.00%	
	(Direct/Indirect)/HOUR					#DIV/0!		\$0.00	#DIV/0!	
RESIN	-					\$0.00		\$0.00	0.00%	
PIGMENT						\$0.000		\$0.000	0.00%	
ADDITIVE	 Dowdor		T			\$0.000		\$0.000	0.00%	
VITROLITE Powder Concentrat				-		\$0.000		\$0.000	0.00%	
TOTAL COST PER CYCLE:					\$0.00		\$0.00	0.00%		
Process Elements				Baseline-Parts		Vitrolite- Parts	Percent Change			
CAVITIES FILLE	D					0		0	0.00%	
CYCLE TIME					0.0		0.0	0.00%		
TOTAL PARTS WEIGHT (GRAMS)					0.0		0.0	0.00%		
PERCENTAGE REJECTS/ HOUR:					0%		0%	0.00%		
CYCLES/HOUR					0.00		0.00	0.00%		
					0.0		0.0	0.00%		
PART WEIGHT (GRAMS) NET PARTS/HOUR:					0.00		0.00	0.00%		
ALL PARTONIO	J					0.00		0.00	0.0070	
				Vitro	olite	® SUMMAR	Y			
PRODUCTIVITY OPPORTUNITY:						0.00%				
PARTS OPPORTUNITY PER HOUR:						0				
						<u> </u>				
Production Improvement Potential/Shift:					Baseline-Parts		Vitrolite- Parts	Benefit		
PARTS PER SHIFT-						-		-	0	
PARTS PER WEEK-						-	•	-	0	
,	/itrolite Required (II	os):			Shift:	- 1	Wk:	- Mo:	_	
					5					

