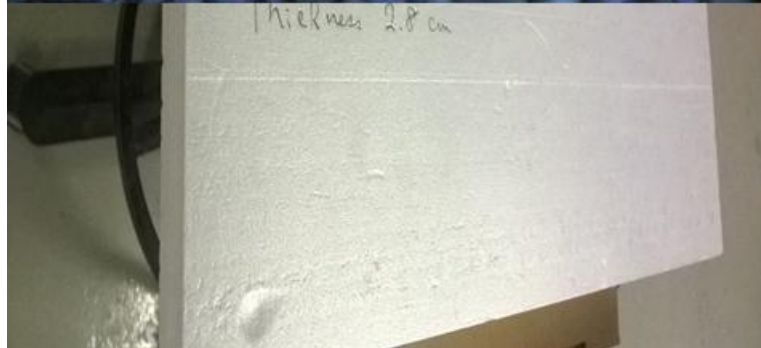
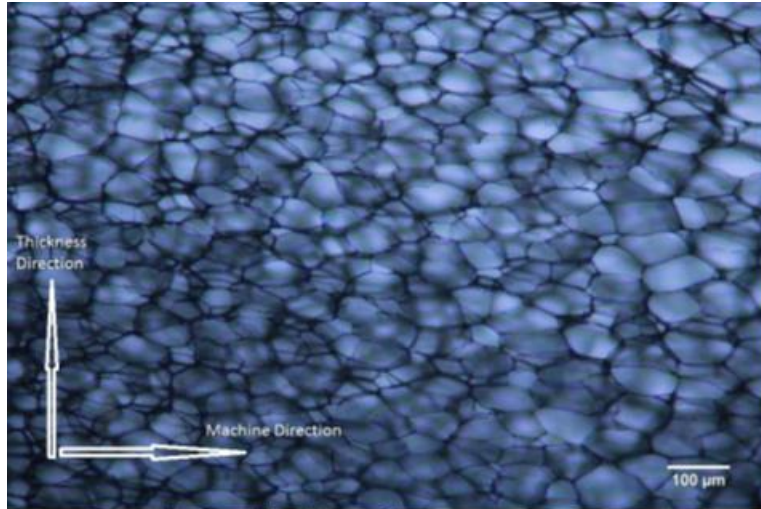


A New Generation of Building Insulation by Foaming Polymer Blend Materials with CO₂

2015 Building Technologies Office Peer Review



Project Summary

Timeline:

Start date: January 1, 2014

Planned end date: December 31, 2015

Key Milestones

1. Insulation value of R-6 per inch, pore geometry and orientation created by the foam extrusion process with 97% porosity (30 kg/m³ density) and full-scale manufacturing costs of < \$0.40/ft²; 12/31/14
2. Insulation value to R-9 to R-10 per inch with foaming density less than 30kg/m³ and full-scale manufacturing costs of < \$0.70/ft²; 12/31/15

Budget:

Total DOE \$ to date: \$244,038

Total future DOE \$: \$165,962

Target Market/Audience:

Residential and commercial building walls and foundations; both retrofit and new construction.

Key Partners:

Armacell International	Hoswell Corporation
---------------------------	------------------------

Project Goal:

Develop a new, environmentally clean building insulation with significantly superior performance but competitive costs on a per R-value basis. The key is to improve pore size, geometry, and orientation to maximize insulation value while keep using the most cost-effective foam extrusion production.

Purpose and Objectives

Problem Statement: Building insulation has not exceeded R-5/inch insulation value at the commercial level due to cost-efficiency and innovation issues. The industry standard is extruded polystyrene (XPS), which is also produced using environmentally harmful HFCs.

Target Market and Audience: The initial target market for our technology is residential and commercial building walls and foundations, which currently utilize older, lower R-value insulations (e.g. XPS at R-4 to R-5/inch). Overall, wasted heat flow in this market consumes over 5 Quads of energy per year. The target audience is existing XPS manufacturers (e.g. Dow, Owens Corning, etc.), as XPS products are produced with environmentally harmful methods and have lower R-values than the project target.

Impact of Project:

1. The target is a high R-value (minimum R-6/inch, maximum R-10/inch) foam insulation for buildings that is cost-competitive on a per-R-value basis.
2. How we are measuring achievement towards goal:
 - a. Near-term: R-value, density and cost targets based on sample measurements and raw materials/processing analysis; signing commercial agreement with partner
 - b. Intermediate-term: Manufacture/sell product that is coded for installation in US
 - c. Long-term: Annual product sales that are 10% of the \$4B foam insulation market, which we had estimated would yield 361 TBTU energy savings

Approach

Approach:

- (1) Developing and commercializing HFC-free building insulation with R-value increased above R-5/inch
- (2) Producing advanced foam insulation with pore morphology control and CO₂ foaming so that R-6/inch foam building insulation can be manufactured with a competitive cost
- (3) Incorporating material nanotechnology to improve foam insulation value to R-7~10/inch at laboratory scale in two years, and subsequently commercializing this new product in the building materials market with a venture-capital supported project from years 3 to 5.

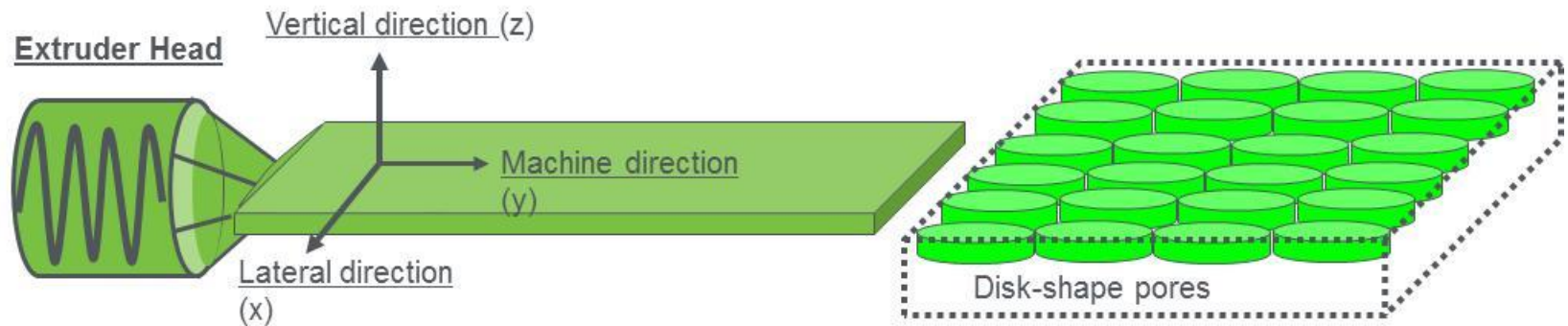
Key Issues:

- (1) Increasing insulation (R/inch) value while reducing the total cost on a per-R-value installed basis
- (2) Directly implementing technologies in most cost-effective foam extrusion product process
- (3) Significantly reduce pore size and control pore geometry in a fast foaming process

Distinctive Characteristics:

- (1) If technology development is successful, the impacts to market and energy savings are immediately accomplishable
- (2) The technology will lead to an advanced insulation with the least material consumption and lowest processing cost

Previous project – Affordable Super Building Insulation by CO₂ foaming process



- First full-capacity factory trial was successfully carried out on October 20-23, 2012 at Hoswell Shanghai insulation plant
- Commercial size insulation boards were produced without using any HFC and the products demonstrated exceptional insulation compared to any samples made by CO₂ blowing in previous experiments
- Aging studies of thermal conductivity and dimensional stability were done in collaboration with Oak Ridge National Lab and Hoswell

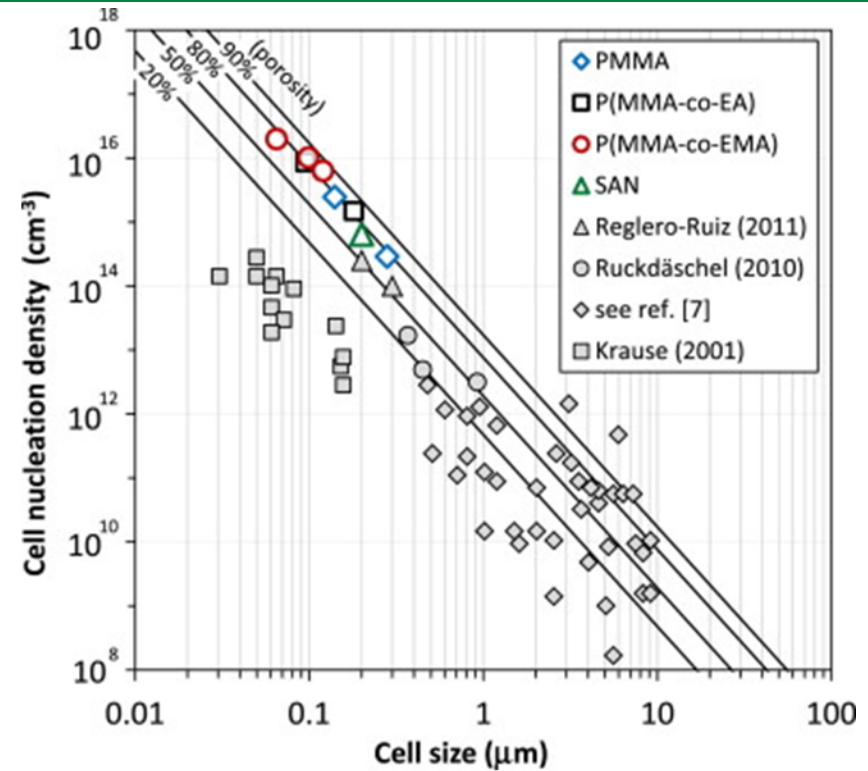
Goal of Making Super-Insulation Foam is Extremely Challenging

$$P_{in} - P_{out} = \Delta P = \frac{2\sigma}{r}$$

$$\Delta G_a = \frac{4\pi r^2 \sigma}{3} \quad \Delta G_a = \frac{16\pi\sigma^3}{3(\Delta P)^2}$$

1st stage – Nucleation and Growth,
110 to 50 bar (as rapid as possible)

2nd stage – Expansion
50 to 1 bar (within 5 second)



σ (dyne/cm)	Nucleus radius ($\Delta p = 2\sigma/r = 60$ bars)	Nuclei density (/cc) (Estimated)	Pore size after growth stage	Pore size after expansion
20	6.67 nm	6×10^8	8 micron	25 micron
15	5 nm	5×10^{15}	32 nm	100 nm

Reducing pore size to submicron while keeping porosity high (>95%) is extremely difficult.

“Low density thermoplastic nanofoams nucleated by nanoparticles”, Stephaney Costeux, Lingbo Zhu, Polymer, Volume 54, Issue 11, 2013, 2785 - 2795

Progress and Accomplishments

Lessons Learned:

Factory trial – Die modification is substantially more difficult at larger scales due to the very high throughput

Factory trial – Incorporation of additives into new polymer blends (rather than pure PS) creates new processing challenges (degradation) that must be overcome

Accomplishments:

Previous project – Developed a cost-effective, commercial-stage R-5 per inch building insulation produced via environmentally clean CO₂ foaming process

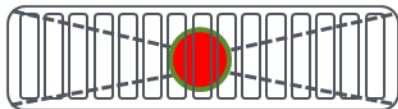
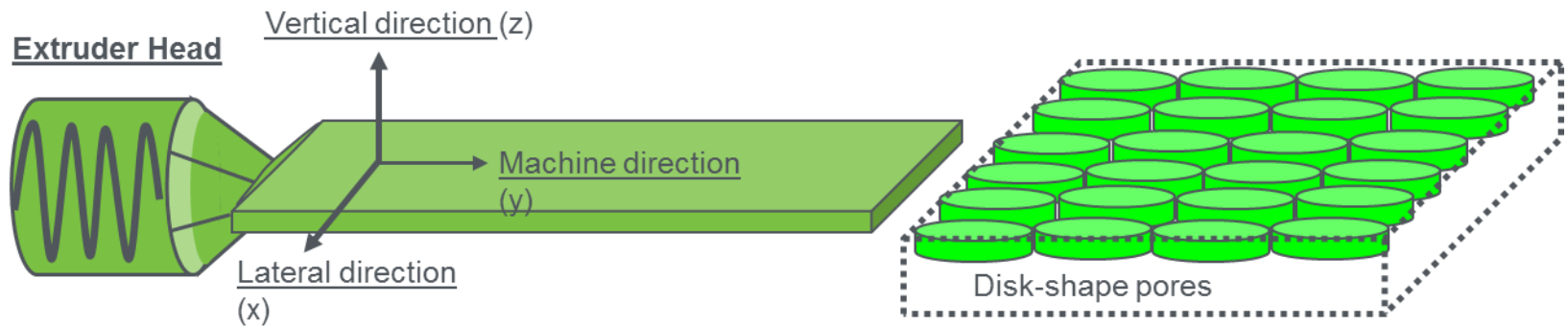
Current project – Successfully foamed new polymer blend insulations that set the stage for improvements to R-6; developed new clay additive technology that can greatly enhance polymer foam properties

Market Impact:

1. We have engaged a commercial partner (Armacell International) to accelerate time-to-market via their expertise on PET materials and assistance in factory scale-up and trials using their facilities
2. We have successfully lowered density and pore size at reasonable cost per-R-value per milestones; R-value TBD based on upcoming trial

New die design patent filed

Extruded insulation foam board normally expands in all three directions; Promoting bubbles' expansion in lateral and machine direction while depressing vertical expansion can produce the favorable oriented, disk-shape pore structure.



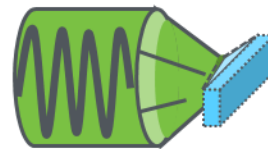
Swelling ratio



Extrudate coming from an orifice through the tapered section into the plural-slit die.

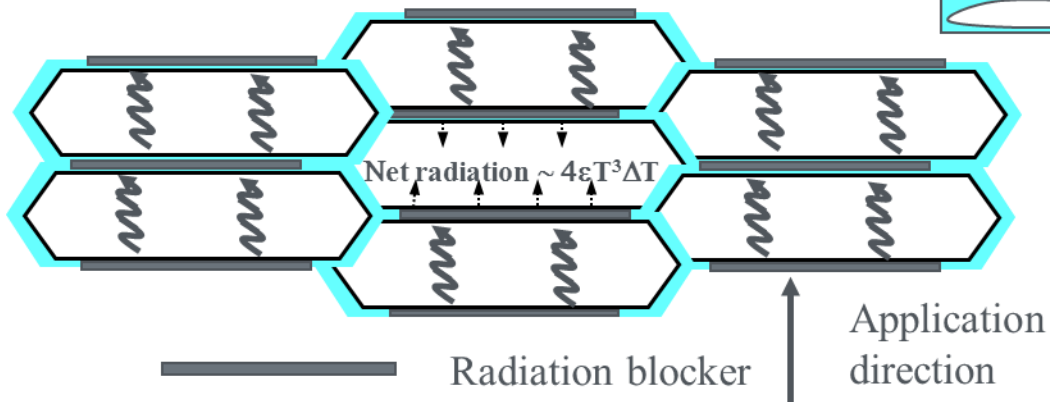
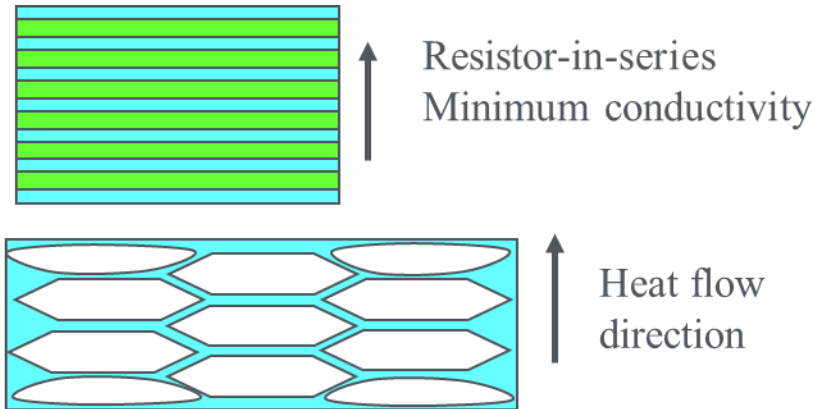


Extruder Head



ISTN technology innovations

(1) Aligning oblate (disc-shaped) pores against heat flow direction to reduce thermal conductivity in application direction – increasing R from R-4.2/inch to R-5.5/inch.



(2) Adding reflective or absorptive radiation blockers in a layered structure can minimize radiation loss – increasing R from R-5.5/inch to R-7/inch.

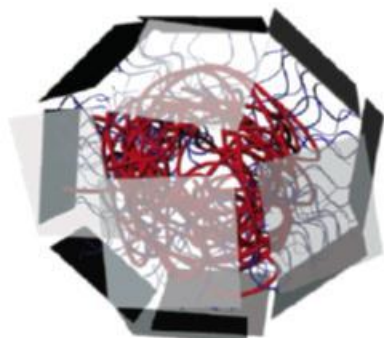
Using exfoliated and aligned clay platelet molecules to confine foaming could potentially achieve both pore size reduction and geometry control.

New Clay Exfoliation Technologies

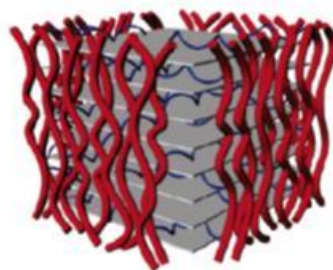
"One-Step Exfoliation of Montmorillonite via Phase Inversion of Amphiphilic Copolymer Emulsion", Chih-Cheng Chou and Jiang-Jen Lin, *Macromolecules* 2005, 38, 230-233

Table 2. XRD d Spacing, Organic Fraction, and Dispersing Size of MPP/POA-Amine Intercalating MMTs

intercalating agent	d spacing ^a (Å)	weight fraction (w/w) ^b		average diameter (nm)	
		based on CEC	based on TGA	25 °C	75 °C
none	12.4	0/100			
at 80 °C ^d					
MPP	12.4	74/26	32/68		
MPP-POE2000	19.5	84/16	82/18	570	580
MPP-POP2000	19.1	84/16	76/24	500	430
at 120 °C ^e					
MPP-POE2000	featureless	84/16	84/16	125	150
MPP-POP2000	featureless	84/16	84/16	115	120



MPP-POE2000/MMT
oil-in-water (OW) type

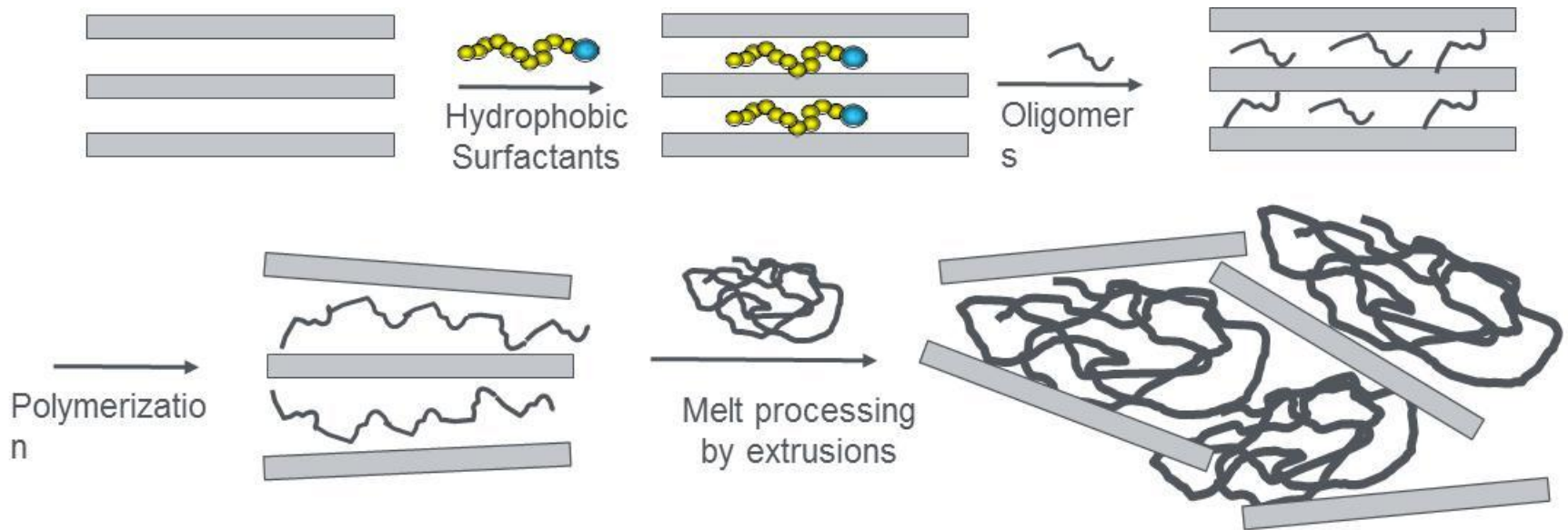


MPP-POE2000/MMT
water-in-oil (OW) type

We plan to foam the polymer within the core and utilize the exfoliated clay to restrict the bubble coalescence.

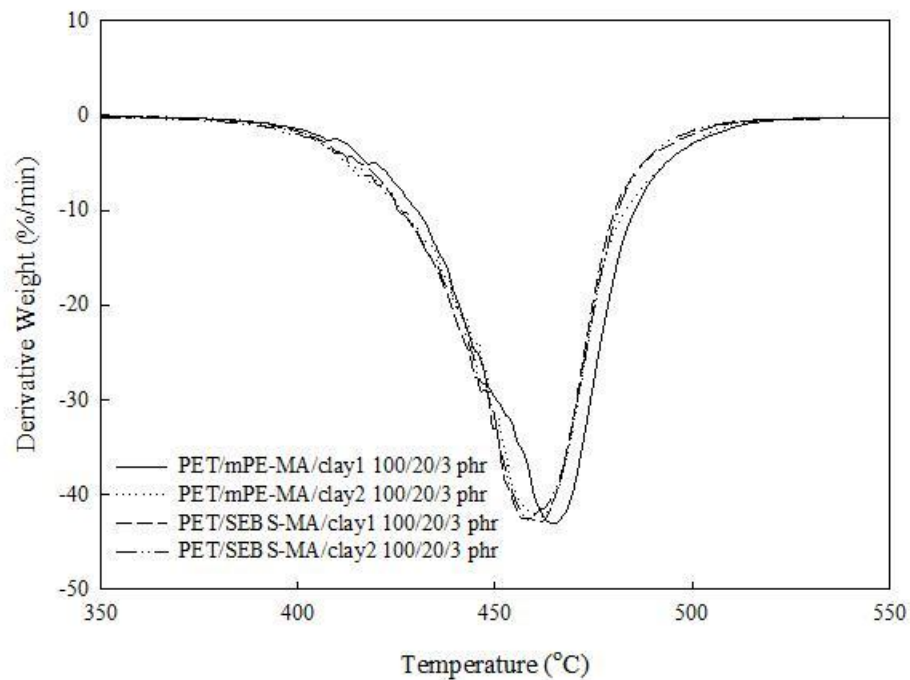
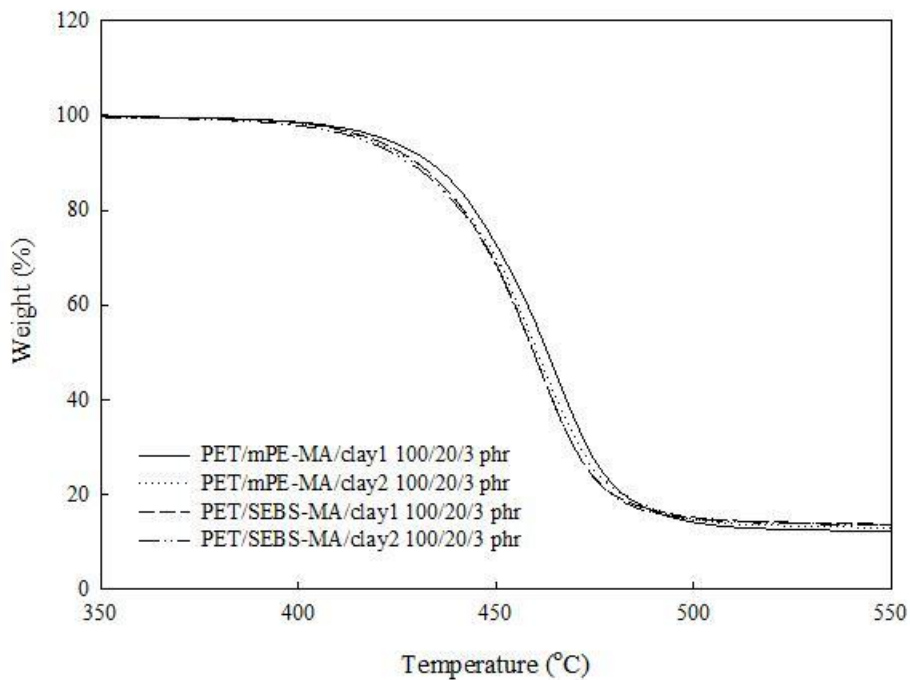
ISTN Simplified processing for better efficiency

- Current processing scheme: Intercalation of oligomers followed by polymerization and compounding



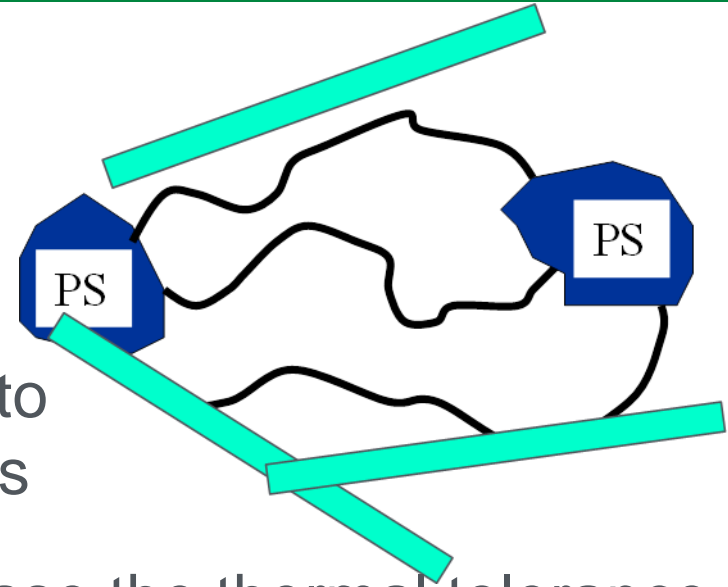
The above is too complicated and expensive for common polymers. ISTN has accomplished a new processing strategy that can directly incorporate silicone polymer (m.w. > 10,000) to exfoliate clay and its composite.

TGA of PET-Clay-Elastomer composite



Blends of PE, PS, SEBS, PET and Clay

- SEBS functions as a rheology modifier and compatibilizer for blend of PE, PS, PET
- Ratio of PE and PS can be utilized to adjust the flexibility of foam products
- PET and Clay can be used to increase the thermal tolerance and temperature range of the foams
- ***Controlling domain size in a polymer blend is a novel approach to reduce pore size by confinement***
- A basis for designing blends of future high-temperature thermal insulation



Importance of foaming PET blend insulation

- Using the blend of elastomers (ma-SEBS/m-PE) and exfoliated clay to improve mechanical performances and lower density of Armacell PET foam
- If the scheduled pilot foaming experiment is successful, this technology will become the most effective approach of improving building insulation value to and above R-6/inch targeted by our project
- In addition, based on preliminary laboratory results, the new technology could serve as a base system for a new generation of high-temperature thermal insulation that has higher temperature range (above 250°C), lower density, and easier installation process
- These tests will also provide valuable proof-of-concept for the silicone-clay technology's ability to enhance foams not only for high-temperature insulation, but other major such as automotive and aviation foams

Project Integration and Collaboration

Project Integration: We have collaborated with two commercial partners, Hoswell and Armacell International, in factory foaming trials to successfully demonstrate our polymer blend foam technology at the production scale. In these collaborations, team members worked with the partners to a) negotiate terms of the commercial relationship and b) execute the production work, which included coordinated sourcing of materials, compounding of master batches, operating/overseeing the extrusion line and analysis of foam sample data.

Partners, Subcontractors, and Collaborators: No subcontracts. From the project outset, we proactively evaluated commercial partners to allow the best opportunity for reaching the market with our product. One such example is Hoswell (Shanghai), which produces foam products and also sells extrusion equipment; we conducted two factory-scale trials using their facilities. We have since established a collaboration with Armacell, which also has significant interest in our technology with the added benefit of expertise in PET materials and production of industrial application foams. Armacell has benefited our production-scale testing, and may license our technology in the future.

Communications: 2013 Emerging Technologies and Building America Workshop.
2014 Peer Review.

Next Steps and Future Plans

Technical:

Upcoming factory trial with Armacell to produce PET blend foams and evaluate properties against project milestones.

Foaming polymer-clay composites in a pressure vessel to determine feasibility of making submicron pore sized blend foams.

Scale up clay production to support larger-scale pilot and factory trials of polymer blend foaming.

Commercial:

Continue licensing discussion with Armacell. Specific terms for PET/building insulation market (i.e. upfront/milestone payments, royalties, exclusivity, etc.). Also determine other markets based on new clay technology (e.g., high-temperature foams, automotive, aviation, etc.)

Developing the polymer-clay nanocomposite business in collaboration with Armacell, Taiwan surfactant and Hoswell.

REFERENCE SLIDES

Project Budget

Project Budget: \$575,000 (\$400,000 DOE / \$175,000 cost-share)

Variances: No variances

Cost to Date: \$348,914 (\$244,038 DOE / \$104,876 cost-share)

Additional Funding: None

Budget History

FY2014 (01/01/15-03/31/15) (past)		Q1 2015 (01/01/15-03/31/15) (current)		Q2 to Q4 2015 (04/01/15-12/31/15) (planned)	
DOE	Cost-share	DOE	Cost-share	DOE	Cost-share
\$244,038	\$104,876	\$48,544	\$20,805	\$107,418	\$49,319

Project Plan and Schedule

Major Task Schedule

Phase	SOPO Task #	Item: Task = T Milestone = M Deliverable = D	Task Title or Milestone/Deliverable Description	Performer (if different from recipient)	Task Completion Date				Progress Notes
					Original Planned	Revised Planned	Actual	% Complete	
1	1.0	T	Task 1.0 Polymer Blend and Composite Technology (PS, PET, clay)				75%		
1	1.1	T	Subtask 1.1 Intercalation of Block-Copolymer into Clay Gallery			03/31/14	100%		
1	1.1	M	Q1 milestone: Demonstrate pore size < 10 micron, foam density < 50 kg/m3.			03/31/14	100%	Better density (~44 kg/m ³), worse pore size (50 micron); combination of density and pore size is most important	
1		D	Research Performance Progress Report (RPPR)			03/31/14	100%	Completed	
1		D	SF-425, Federal Financial Report			03/31/14	100%	Completed	
1	1.2	T	Subtask 1.2 Foaming of a Polymer Blend of PS, SEBS, and PET			06/30/14	100%		
1	1.2	M	Q2 milestone: Demonstrate (PS + SEBS + Clay) pore size < 10 micron and foam density of 30 ~ 40 kg/m3.			06/30/14	100%	Good; density lower than goal at 30 ~ 35 kg/m ³ , although pore size remains a challenge	
1		D	Research Performance Progress Report (RPPR)			06/30/14	100%	Completed	
1		D	SF-425, Federal Financial Report			06/30/14	100%	Completed	
1	1.3	T	Subtask 1.3 Foaming of PET-Clay Composite			09/30/14	100%	Completed	

Project Plan and Schedule

Phase	SOPO Task #	Item: Task = T Milestone = M Deliverable = D	Task Title or Milestone/Deliverable Description	Performer (if different from recipient)	Task Completion Date				Progress Notes
					Original Planned	Revised Planned	Actual	% Complete	
1	1.3	T	Subtask 1.3 Foaming of PET-Clay Composite		09/30/14		09/30/14	100%	Completed
1	1.3	M	Q3 milestone: Demonstrate PET foam density of 40 kg/m ³ .		09/30/14		09/30/14	100%	Density range of 29.7-31.2 kg/m ³ ; increased m-PE content led to higher density
1		D	Research Performance Progress Report (RPPR)		09/30/14		09/30/14	100%	Completed
1		D	SF-425, Federal Financial Report		09/30/14		09/30/14	100%	Completed
1	1.4	T	Subtask 1.4 Production of R-6 per inch Building Insulation using PS/PET/Clay Material Composites and CO ₂ blowing agent		12/31/14	03/31/15		50%	In-process; Armacell trial delayed to Feb 2015
1	1.4	M	PHASE 1 GO/NO-GO MILESTONE (Q4): The decision of continuation will be based on three deliverables using the results of a production-scale extrusion line: (a) insulation value of CO ₂ blown foam above R-6 per inch, (b) pore geometry and orientation created by the foam extrusion process with 97% porosity (30 kg/m ³ density) and (c) full-scale manufacturing costs of < \$0.40/ft ² .		12/31/14	03/31/15		67%	Achieved 97% porosity and 30k/m ³ density, achieved < \$0.40/ft ² cost target, R-6 per inch dependent on upcoming trial
1		D	Research Performance Progress Report (RPPR)		12/31/14		12/31/14	100%	
1		D	SF-425, Federal Financial Report		12/31/14		12/31/14	100%	
1		D	Special Status Report		12/31/14				