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ISTN

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1. Executive Summary

Background

Thermal building insulation is among the most important solutions to the global energy crisis. According to the U.S. Energy Information Administration, new energy generation requires an investment of $3,362/kW for a fossil-energy plant, and $5,530/kW for a nuclear power plant. In contrast, the application of thermal building insulation reduces energy consumption at an investment of only $125/kW. This low cost highlights the importance of insulation materials in building structures, which are responsible for 40% of global energy consumption and 24% of carbon dioxide ("CO_2") emissions. Given such intensive energy use, an improvement in the thermal resistance, or R-value [a], of building insulation would reduce energy waste at a considerable level. However, the insulation industry is notoriously conservative and has not developed commercial R-value improvements in many decades. The current "2nd" generation of thermal insulation technology is almost 70 years old. A breakthrough in advanced materials would benefit energy efficiency significantly in the U.S., as well as developing markets such as China and India, where many buildings are poorly insulated despite rapidly growing populations and energy consumption.

The insulation industry's lack of innovation has created opportunities for technology disruption in several key market segments, such as extruded polystyrene ("XPS") [b]. XPS comprises a $5.1 billion worldwide market with two major needs: 1) higher R-value foams without cost increase, and 2) new foaming agents to replace environmentally damaging hydrofluorocarbons ("HFCs") in the production process. Improvement of R-values is a significant technological challenge because the current processing methods and raw materials mix have been optimized over many decades to produce the best R-value at the lowest cost. Also, while HFCs are necessary for high R-value, they have 1,300 times the global warming power of CO_2 – a risk that has driven numerous changes in international environmental policy, including bans of HFCs in many of the major industrialized countries over the next few years. These bans are a catalyst for significant increases in commercial demand of environmentally clean XPS, and would benefit early entrants that could establish a strategic market position for the introduction of higher R-value alternatives that significantly enhance future energy efficiency. The prevailing problem for existing manufacturers has been that the conversion to a CO_2 foaming process would result in a trade-off of approximately 20% lower R-values than the current industry standard in HFC-foamed products.

In 2010, ISTN received a DOE ARRA grant award to develop advanced building insulation (DE-EE0003983) that solves the problems of replacing HFCs with clean blowing agents at competitive performance and cost. During the three-year project, we developed a new XPS building insulation with CO_2 as a clean foaming agent to replace HFCs, while achieving competitive R-value and production costs: full-scale factory manufacturing trials resulted in R-5/inch and costs of less than $0.35/board-ft. [c]. Key innovations included the superior control of foam cell size, geometry and

a R-value (thermal resistance) is a common measure of insulation value, equivalent to the reciprocal of thermal conductivity in units of $ft.^2 \cdot °F \cdot hr./Btu$. R-5/inch insulation of 3-inch thickness has total R-15. R-values in this report based on temp. of 75°F.

b XPS is a rigid foam board with widespread use in wall sheathing and foundational components due to high R-value, rigid structure, and cost-efficiency. The manufacturing process uses extruders to expand a molten PS resin with HFC foaming agents into sheets of low density, closed cell foam. XPS annual sales are $1B in the U.S. (12% of U.S. market) and $5B WW.

c $/R/board-ft. is the relevant metric for comparing insulation costs on an R-value adjusted, per unit basis. R-5/inch insulation that costs $0.35/board-ft. has an R-value adjusted cost of $0.07/R/board-ft. A board-ft. is 12 in. x 12 in. with 1-inch thickness.

orientation, which is substantially beneficial to R-value and cost-efficiency. The work in that project set the stage for further nanotechnology advancements in insulation materials that could lead to R-values 50% greater than the current state-of-the-art.

Project overview

In this project, ISTN proposed to develop a new, environmentally clean building insulation with the potential for superior performance (R-9 to R-10 per inch) to existing insulations and our previous CO_2-blown foam (R-5 per inch), as well as competitive costs per R-value (<$0.70/board-ft.). This new technology builds off of the previously developed platform by continuing to use CO_2 in place of HFCs as an environmentally clean blowing agent, and implementing the superior control of porosity on the production line. In addition, the key differentiators of the newly proposed technology were the incorporation of polymer blends and new composites in place of the polystyrene ("PS") resin traditionally used in XPS production, and introducing secondary nanostructures to further boost insulation value. The end target was a cost-effective process that could be used to manufacture a competitive product to XPS, with significantly higher R-values than the current industry standard of R-5 per inch.

		Thickness	R-value		Price	
Material type	Category	(inches)	Total	Per inch	$/board-ft.	$/R/board-ft.
Fiberglass batt	Fiberglass	3.5	13.0	3.7	$0.20 – $0.40	$0.02
		12.0	30.0	2.5	$0.60 – $1.00	$0.03
Loose fill (fiberglass, cellulose, and mineral wool)	Fiberglass	8.0	30.0	3.8	$0.45 – $1.35	$0.03
		23.0	50.0	2.2	$0.75 – $2.25	$0.03
Polyurethane spray foam – open cell	Foamed polymer	3.5	12.6	3.6	$1.70 – $2.50	$0.17
Polyurethane spray foam – closed cell	Foamed polymer	1.0	6.5	6.5	$1.30 – $2.00	$0.25
EPS (expanded polystyrene) foam board	Foamed polymer	1.0	4.0	4.0	$0.20 – $0.35	$0.07
Polyisocyanurate foam board	Foamed polymer	1.0	6.5	6.5	$0.60 – $0.70	$0.10
XPS (extruded polystyrene) foam board		**1.0**	**5.0**	**5.0**	**$0.40 – $0.55**	**$0.10**

Table 1. Comparison of state-of-the-art insulation products.

In the U.S., state-of-the-art XPS products are primarily manufactured by two companies: Dow and Owens Corning. Although these products (i.e., STYROFOAM and FOAMULAR) have a starting thermal resistance of R-5.0/inch, the R-value declines over the life of the product as the HFC blowing agents essential to high R-value exchange with air in the environment. In the existing technologies, the substitution of CO_2 for HFCs as the primary foaming agent results in a much lower starting R-value, as evidenced in CO_2-foamed varieties of XPS in Europe with R-4.2 per inch insulation value. Our previous project had achieved a cost-efficient, CO_2-foamed product of R-5.2 per inch, able to be produced on a full-scale extrusion line. The goals of our new proposal were to continue the improvement trend and further raise insulation value to R-6 per inch, and then R-9 to R-10 per inch with the incorporation of advancing material nanotechnology.

The major achievements from this project were ISTN's development of a new product that can achieve thermal resistance of up to R-6 per inch (20% better than the best existing products), and a novel additive that is the first realistic option for creating R-9/inch foams using the cost-effective extrusion process. The high throughput of this process and our choices of materials also ensure production costs on a per R unit basis that are less than the cost of Dow and Owens Corning XPS products.

From the beginning, this project was designed to create a new platform for super-thermal, nanopore insulation using novel innovations that are applicable to the foam extrusion process utilized in XPS manufacturing, giving high-throughput production and optimal cost-efficiency.

Similar to our previous project, the design included the use of CO_2 as a clean blowing agent to replace HFCs, which will be phased out in Europe and the U.S. by 2020 and 2021 respectively. Unlike any previous attempts (including our own prior project), the performance goal of our new innovations was to greatly exceed the long-standing thermal resistance barrier of R-5 per inch in XPS (with benefits of entrapped HFC) and similar, extruded products, a level that had held constant for over three decades. We were able to demonstrate a meaningful improvement beyond this threshold, and most importantly do so with cost-effective production processes, as a previous problem comment to super-thermal nanopore insulations (i.e., aerogels) was the dependency on batch production processes with exceedingly high costs. By accomplishing the major goal of a higher performance building insulation product, this project was able to create a product and platform that meets DOE goals of enhancing U.S. energy efficiency, sustainability and manufacturing job opportunities.

Technical goals and accomplishments

There were three main goals of ISTN's project:

(1) Next-generation polymer blend foams – Developing advanced material composites with pore morphology control and CO_2 foaming to achieve R-6 per inch foam building insulation at a cost of <$0.40/board-ft.;

(2) Super-insulation foam platform – Incorporating nanotechnology structures and materials that will significantly improve upon R-6 by reaching foam insulation values of R-9 to R-10 per inch up to the pilot scale (manufacturing cost <$0.70/board-ft.);

(3) Commercialization – Laying the groundwork for commercialization of a new insulation venture after the project, including detailed strategy and analysis, and the evaluation of industrial and/or venture-capital partners.

Our technical plan was to develop the next-generation polymer blends (1) within Year 1 of the project, which would then serve as the foundation for the nanostructure enhancements and more advanced R-value improvement work for developing the super-insulation platform (2) over Year 2 of the project. In parallel, commercialization activities would be conducted in both Years 1 and Year 2. ISTN's major innovation was the development of production-level foaming of blends of polymers and inorganic nanostructured additives to substantially enhance thermal insulation performance of XPS while maintaining the new products' cost competitive to conventional building insulations. Major technical accomplishments of the project are summarized below:

(1) We had successfully demonstrated at the manufacturing scale that a blend of PS with other polymers (for example, SEBS, m-PE, etc.) and inorganic silicates can be foamed with predominantly CO_2 to the low densities (~ 30 kg/m^3) required by commercial foam insulation.

(2) At both the pressure vessel as well as pilot extrusion levels, we designed cost-efficient recipes for a new generation of building insulation product which can achieve R-6/in performance with blends of Polymer-Blend-A [d], SEBS-MA and nanoclay. The breakthrough in clay exfoliation by creating and using novel silicone polymer surfactants was the key to lowering cost, with the additional benefits of increasing performances at elevated temperatures.

(3) The invention of integrating the supercritical drying of nanostructured inorganics with the low-

d Polymer-Blend-A refers to a group of materials that are kept confidential in this report. We originally planned for this to be one specific polymer in our proposal, but over the course of the project realized the need for changes and instead having a blend.

cost CO_2 foaming of polymer blends can produce secondary nanostructures by a foam extrusion process and thus is capable of further increasing future building insulation performance to R-8/inch without prohibitive cost increase.

A summary of key product performance enhancement options concluded from this project are included below:

Insulation Enhancement	R-4.2 to 5/inch	R-5 to 6/inch	R-6 to 7/inch	R-7 to 8/inch
Technology	Radiation inhibition, cell geometry control	PET-clay based, density ≈ 40 kg/m^3, cell ~ 10 μ	Low level (<20%) Secondary nanostructure	High level (>50%) Secondary nanostructure
Cost factor	Low wt. % additives	Density reduction	Integration of drying with foam extrusion	Removing excessive solvent

Table 2. Summary of product performance.

Energy, environmental and economic benefits

Our technical achievement in producing insulation with the highest R-value via CO_2 foaming will allow all building insulation manufacturing to replace the use of HCFCs and HFCs, generating a huge environmental benefit of eliminating both ozone depleting and high global warming power emissions. The below table compares the global warming power of CO_2 to the most common blowing agents for XPS. As an example, blowing agents comprise an average of 15% of XPS materials, so 1 metric ton of today's XPS would require approximately 150kg of HFC-134a. This total has an emissions impact of 195,000 kg of CO_2. In contrast, a CO_2-foamed product would have an emissions impact of only 150 kg (i.e., a 99.9% reduction in emissions).

Source	CO_2	HFC-134a	HCFC-142b
GWP100	1	1,300	2,000

Table 3. Global Warming Potential ("GWP") Values [1].

In a 100% market penetration case, we calculate energy savings of 2.8 Quads per year, 262 million metric tons of CO2 emissions savings per year, and $71.5 billion in economic savings. As a baseline commercialization case, we modeled 12% penetration of the U.S. building insulation market within 10 years. This assumes our commercialization of an R-5 per inch clean insulation product as developed in our previous project, followed by the gradual introduction of R-6 per inch and higher foams as developed in the current project. In this case, our products would provide domestic energy savings of 341 trillion BTU per year. CO_2 emissions savings would be 31.7 million metric tons per year, with 20.4 million metric tons due to the reduced energy waste, and 11.2 million metric tons due to replacing HFCs with CO_2 in foaming production. The emissions savings equate to removing 6.7 million cars from the road or 2.9 million fewer homes consuming energy. Overall, the total annual economic savings would be $8.7 billion, with $7.5 billion from reduced energy waste and $1.2 billion from reduced CO_2 emissions.

Commercialization

Over a two-year project timeline, our proposed objectives and development work led to the creation of an advanced platform for super-thermal foam insulation, to complement our previously developed technology of an environmentally clean XPS product. Based on our commercialization work in this project, we now plan to use the technology portfolio to start a new commercial insulation venture. The team plans to start a production facility for developing, manufacturing and selling CO_2-blown XPS foam, followed by other new foam technologies. The facility's initial production capacity will be less than 2% of U.S. XPS demand, intended to serve a regional footprint due to the high sensitivity of insulation profitability to shipping costs. Future growth strategy will entail the addition of production lines, followed by new manufacturing plants in other regions of North America and key international markets such as China, India and the Middle East. We will also evaluate licensing partnerships in these markets.

Technology	Application	Launch	R-value per inch	Density (kg/m^3)	Price ($/board-ft.)
ISTN-developed products:					
1st-Gen. CO_2-foamed XPS board	Building wall, roof, foundation	2H 2016	R-5	40	$0.45
2nd-Gen. CO_2-foamed polymer blend board	Building wall, roof, foundation	2019	R-7	35	$0.60
Advanced high-temperature flexible foam	Industrial pipe	2021	400°C: R-3	200	$9.00
Other foams produced via extrusion:					
PE/EPE foam sheet	Packaging foam				
PET foam board	Wind turbine blades				
Medium density extruded foam roller	Physical therapy and yoga mats				

Table 4. Commercialization technology pipeline. Technologies developed by the team are planned as three staged commercial product categories. In addition, the foam extrusion line allows production of other common foams such as packing and wind turbine blades, which could serve as additional commercial products.

2. Technical Background

As early as half a century ago, researchers were aware that a composite material's thermal conductivity can be modeled based on component morphology and orientation [2]. This understanding is meaningful for foam insulation, which is fundamentally a composite of solid and air bubbles. As in any composite, the foam's thermal conductivity has an upper limit when both components' thermal conductions are aligned in parallel, and a lower limit when they are aligned in series [3]. Further modeling calculations [4,5] confirm that aligning ellipsoidal inclusions along their longest axis effectively increases thermal resistance in a vertical direction at the expense of resistance in the direction parallel to the long axis. Figure 1 illustrates an example of enhancing insulation value in the direction of application (i.e., temperature gradient direction). Moreover, theoretical calculations also indicate that the alignment of oblate (disk-shaped) cells is even more effective in insulation enhancement than needle-shaped cells [6]. Thus, optimizing the creation and orientation of anisotropic cells in thermal insulation foam boards can maximize R-value enhancement.

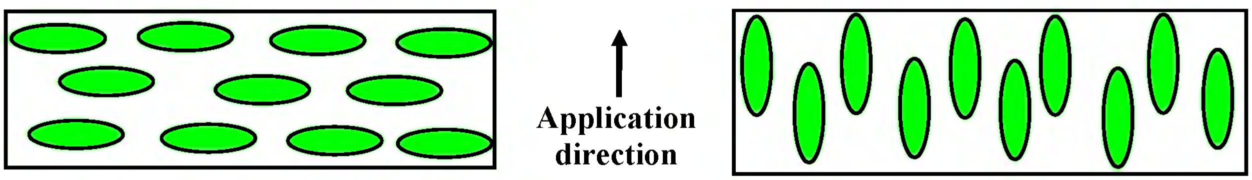

Figure 1. Spheroids aligned in-series (left), or in-parallel with the application direction (right). The left example (in-series) has a lower thermal conductivity (higher insulation value, or thermal resistance) than that of the right.

Realizing these enhancements in the foam production process is a technical challenge. A commercially viable insulation foam with good mechanical properties and cost-efficiency requires a high throughput extrusion process using both high pressure and high temperature. However, only a limited number of materials and process conditions are appropriate for satisfying all of these parameters. Attempts to improve on current commercial XPS insulation require making changes to this delicate balance, and have thus far been unsuccessful.

In our work to develop better XPS insulation, achieving the desired cell orientation and morphology necessitated engineering of the extrusion process to favor the gas bubble expansion in two directions, which are the machine (extrusion) direction and lateral direction (this concept is illustrated in Figure 2). Our design for the art of creating oriented, oblate cells in an extrusion process consists of three procedures: (1) inducing homogeneous nucleation, (2) depressing the die swelling ratio in the vertical direction (z), and (3) facilitating bubble growth in both machine and lateral directions (y, x) via a created tension stress field. We filed a provisional patent application in 2015 [7] to claim these innovations.

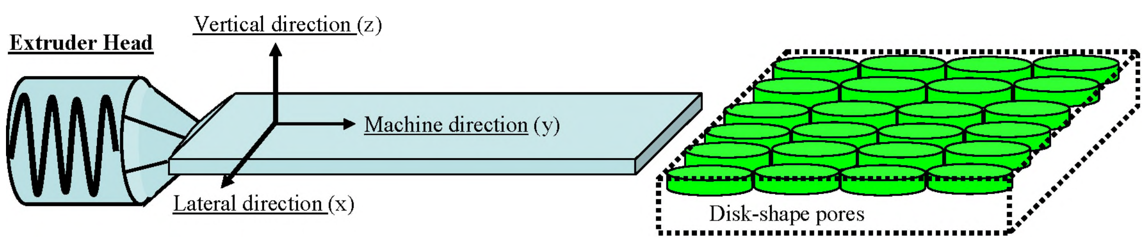

Figure 2. Extrusion process diagram. Promoting expansion in both lateral and machine directions while depressing the vertical expansion leads to oriented cell geometry in favor of the insulation value in vertical direction.

Key innovations in previous project carry over to this project

ISTN's R&D work in factory-scale production experiments has demonstrated the feasibility of manufacturing an XPS building insulation by low-cost foam extrusion process, with control of both cell structure and orientation. The thermal resistance of the new foam insulation has already reached R-5/inch (better than current XPS products that require HFCs to reach the same value of R-5/inch [[e]]), while using CO_2 as the foaming agent to replace HFCs. Additionally, our most recent DOE grant work has demonstrated promising lab-scale results that will lead to further R-value enhancements to create a new industry standard. The key technical innovations we have achieved are:

(1) Controlling size, geometry and orientation of cells – A series of additive and processing technologies were developed to allow active control of the cell size, geometry, and orientation in the post-die foaming of extruded polymer melts. Modified clay additives have been utilized to enhance homogeneous nucleation (for reducing cell size) and induce anisotropic alignment under die shearing stress (for controlling cell geometry and orientation). A new processing scheme to exfoliate clay and intercalate polymer was developed to lower the cost of making polymer-clay nanocomposites.

(2) Integration with foam extrusion process – A key to successfully commercializing an advanced insulation product is to keep the cost of generating porosity (95% of total volume) as low as that of current insulation materials. Based on ISTN's previous experimental results of foam extrusion, we integrated the scheme of raising thermal resistance through controlling cell size and morphology into a continuous foam extrusion production to minimize additional processing cost. This included a novel die design which together with new additive and polymer blend technologies maintains combined material and processing costs at a level that matches fully-installed cost per R-value currently accepted by the market.

In this project, additional innovations were developed to further enhance the product beyond R-5/inch. These innovations include:

(3) Polymer blend and composite technology – Advancements in material nanotechnology have led to the advent of many novel material designs, particularly in the areas of block copolymers and their nanocomposites. ISTN has developed blending and compounding technologies for producing composites consisting of polystyrene, PE, Polymer-Blend-A, and clay uniformly dispersed into domains of submicron scales. We anticipate this type of polymer-clay composite can also be foamed into insulation with enhanced R-per-inch value and improved fire resistance. The scaled-up production of these composites will be engineered and optimized to assure that processing costs remain competitive on a per R-value basis to present insulation base materials. Foaming tests of these new composites are being conducted with the extrusion experiments described above.

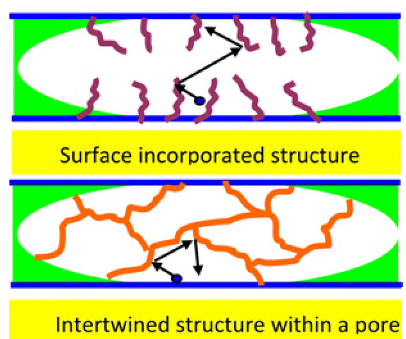

Figure 3. Two secondary nanostructures to reduce heat transfer by air molecules.

(4) Secondary nanostructure to enhance insulation value – We are currently making polymer-clay

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e XPS tends to off-gas HFCs in exchange for air after initial production. The loss of HFCs lowers insulation value to ~R-4.5/inch. Despite this, manufacturers overstate insulation value by advertising R-5/inch, with a product warranty for 90% (R-4.5).

nanocomposites using a blend of block-copolymers, and have developed a scheme of foaming the block-copolymer domains of the composite into a nanostructure containing smaller cells within a primary cell as shown in Figure 3. Exfoliation of the clay platelet molecules and the intercalation of block-copolymer into the gallery of clay platelets are two critical steps of achieving the proposed secondary structure. We are also designing new processing schemes to substantially lower the cost of clay exfoliation and the subsequent intercalation of block-copolymers so that the performance to cost ratio of future, higher R-value materials is justifiable on the cost per R-value installed basis.

3. Project Objectives

Project Scope

In this project, we planned to develop a new, environmentally clean building insulation with superior performance (up to R-9 to R-10 per inch) to existing insulation, as well as competitive costs per R-value (< $0.70/ft^2). The key differentiators of this technology included utilizing CO_2 in place of HFCs as the blowing agent, incorporating polymer blends and new composites in place of traditional polystyrene ("PS"), and introducing secondary nanostructures to further boost insulation value. This technology represents a highly valuable market opportunity given its ability to achieve maximum energy savings (at equal or lower cost) across a variety of thermal insulating applications, such as building foundations and walls, as well as refrigeration and HVAC applications.

The specific aims of this project were:

(1) Next-generation polymer blend foams – Developing advanced material composites with pore morphology control and CO_2 foaming to achieve R-6 per inch foam building insulation at a cost of <$0.40/board-ft.;

(2) Super-insulation foam platform – Incorporating nanotechnology structures and materials that will significantly improve upon R-6 by reaching foam insulation values of R-9 to R-10 per inch up to the pilot scale (manufacturing cost <$0.70/board-ft.);

(3) Commercialization – Laying the groundwork for commercialization of a new insulation venture after the project, including detailed strategy and analysis, and the evaluation of industrial and/or venture-capital partners.

Project Tasks

BUDGET PERIOD 1

Task 1.0 Polymer Blend and Composite Technology (PS, Polymer-Blend-A, clay)

Develop blending and compounding technologies for producing composites consisting of polystyrene, Polymer-Blend-A, and clay uniformly dispersed into domains of submicron scales. Demonstrate foamed polymer-clay composites with enhanced R-value per inch and improved fire resistance. Perform foaming tests of these new composites on production-scale extrusion equipment to verify the low density and scalability of the technology improvements, which will provide an important foundation for further development in Task 2.0.

Subtask 1.1 Intercalation of Block-Copolymer into Clay Gallery

Incorporate new block-copolymer surfactants into a clay gallery to facilitate homogeneous nucleation.

Q1 milestone: Demonstrate cell size < 10 micron, foam density < 50 kg/m^3.

Subtask 1.2 Foaming of a Polymer Blend of PS, SEBS, and Polymer-Blend-A

Incorporate new polymer surfactant into a polymer blend to reduce foam cell size. Test new die design by employing at pilot scale with PS, polystyrene-*b*-poly(ethylene-*co*-butylene)-*b*-polystyrene (SEBS) and clay to lower foam density, and measuring results.

Q2 milestone: Demonstrate (PS + SEBS + Clay) cell size < 10 micron and foam density of 30 ~ 40 kg/m^3.

Subtask 1.3 Foaming of Polymer-Blend-A-Clay Composite

Test new die design by foaming Polymer-Blend-A and Polymer-Blend-A-clay composites at pilot scale and measuring results.

Q3 milestone: Demonstrate Polymer-Blend-A foam density of 40 kg/m^3.

Subtask 1.4 Production of R-6 per inch Building Insulation using PS/POLYMER-BLEND-A/Clay Material Composites and CO_2 blowing agent

Test optimized composites derived from the above three tasks at production scale in order to achieve the lowest density and highest R-value per inch possible.

Q4 milestone: 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) cell 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$.

BUDGET PERIOD 2

Task 2.0 Secondary Nanostructure to Enhance Insulation Value

Fabricate polymer-clay nanocomposites using a blend of block-copolymers. Develop an approach of foaming the block-copolymer domains of the composite into a nanostructure containing smaller cells within a primary cell.

Subtask 2.1 Intercalation of Chemical Foaming Agent within Clay Molecules

Initiate foaming by a chemical foaming agent intercalated within the clay gallery area with the objective of further reducing cell size through homogeneous nucleation.

Q5 milestone: Demonstrate polymer-clay foam with cell size of ~1 micron.

Subtask 2.2 Foaming Nanocomposite of Clay and Block-Copolymers

Intercalate block-copolymers within clay molecules in the gallery region in order to form secondary structure during foaming with a goal of reducing secondary cell size to between 100 nm to 1 micron.

Q6 milestone: Copolymer-clay composite with R-value > R-8 per inch and foam density less than 30 kg/m^3.

Subtask 2.3 Intertwined Secondary Nanostructure

Create an intertwined secondary nanostructure within larger cells of a low-density foam to further increase its R-value to that of a super insulation provided that the structure created does not increase foam density.

Q7 milestone: Create a foam with intertwined secondary nanostructures with preliminary R value >8 per inch.

Q8 milestone: Insulation value to R-9 to R-10 per inch with foaming density less than 30kg/m^3 and full-scale manufacturing costs of < $0.70/$ft^2$.

ALL BUDGET PERIODS

Task 3.0 Technology-to-Market Strategy

Develop a strategy for commercializing this new product in the building materials market with a venture-capital supported project from years 3 to 5. The necessary analysis of IP, value proposition and market viability will be performed in order to add an appropriate commercialization partner. The technology's economic primary energy savings will be included in the BTO prioritization tool and updated based on actual achieved performance and cost estimates.

Subtask 3.1 Market Analysis

Perform analysis of IP landscape. Research initial target markets for the new insulation technology. Develop strategy for each potential market.

Q2 milestone: 3-page report to include (a) assessment of IP landscape (precedents, competitors, opportunities), (b) summary of initial target markets (market dynamics, key companies, size of market opportunity), and (c) preliminary commercial strategy for each market (optimal partner(s), value proposition).

Subtask 3.2 Commercial Partner

Based on market analysis, identify broad list of companies and venture capital firms as potential commercialization partners and begin outreach process to find the best partner for the new technology.

Q3 milestone: 2-page report identifying top three potential industrial partners and top three potential VC partners, with relevant business metrics, fund details, rationale for each partner and summary of discussions to date.

PHASE 1 GO/NO-GO MILESTONE (Q4): Complete NDA and/or MOU, and obtain official letter of interest with at least one industrial or VC partner.

Q7 milestone: If industrial partner: have a draft licensing agreement in place. If VC partner: have a draft investment term sheet in place.

Subtask 3.3 Intellectual Property

File provisional patents based on technology developed in the proposed work and preceding analysis of IP landscape.

Q5 milestone: At least one provisional patent filing for technology from this project.

Subtask 3.4 Manufacturing and Sales Strategy

With commercial partner, develop strategy for new technology to be manufactured in the US. Also with commercial partner, identify best product distribution strategy for initial target markets.

Q6 milestone: 10-page business plan including (a) technology overview, (b) market overview, (c) manufacturing strategy and related capital requirements, (d) distribution strategy and related capital requirements.

4. Project Results and Discussion

A. Task 1.0 Polymer Blend and Composite Technology

Subtask 1.1 Intercalation of Block-Copolymer into Clay Gallery

Significant accomplishments

Completed subtask 1.1 by performing insulation foaming trial (using ISTN recipes) at the Fraunhofer Institute for Chemical Technology in Pfinztal, Germany as part of an existing ISTN collaboration with E-Company [f], a world leader in specialty chemicals. The trial employed a KraussMaffei Schaumtandex laboratory extruder unit (ZE30/KE60) over one day of run-time, during which we produced 1500 board ft. of insulation. Relative to our Q1 milestone of demonstrating foam density <50 kg/m^3 and cell size < 10 micron, we achieved significantly better density (best of 44kg/m^3) and slightly higher than desired cell size (best of 50 micron). Overall, the results were extremely positive as the combination of cell size and density are the key driver in enhancing R-value.

Detailed discussion

We completed subtask 1.1 by performing an insulation foaming trial at the Fraunhofer Institute for Chemical Technology in Pfinztal, Germany as part of an existing ISTN collaboration with E-Company International Ltd., a world leader in specialty chemicals. E-Company had previously had an interest in licensing ISTN's R-5 insulation technology, and the ongoing discussion and collaboration allowed us to evaluate their fit as a long-term commercial partner.

For this trial, we prepared 2 proprietary recipes of materials to foam using a twin-screw extruder (ZE30) as a primary extruder (plastification, mixing nucleating agents and injection of a blend of carbon dioxide and ethanol as physical blowing agents) and a secondary single-screw extruder (KE60) for cooling and homogenizing the melt. The feed rate was 40 kg/h. The melt was foamed through a slot die and then fed into a sheet calibrator. ISTN personnel collaborated with E-Company and Fraunhofer staff on the production line. The trial took place over one day of run-time, during which we produced 1500 board ft. of insulation.

Run	Nucleating Additive	ISTN Polymer Blend	Board Thickness (mm)	Mean Cell Diameter (µm)	Density (kg/m³)
601	Talc	none	31	214	48
629	Talc	7%	31	162	47
635	CBA	7%	18	62	48
636	CBA	12%	20	52	44

Table 5. Selected results from foaming trial.

f One of multiple Europe-based companies with which we collaborated on the project. Actual company name not shown in this report for confidentiality purposes.

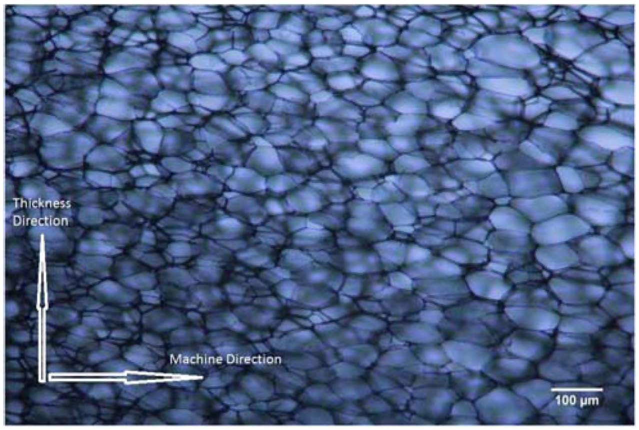


Figure 4. Transmission Optical Photomicrograph of Polystyrene Foam Run 636.

With the combination of a standard nucleating agent (talc) and ISTN polymer blend, the cell diameter reduced from 214 to 162 μm (comparison of run 601 with 629). A chemical blowing agent (CBA) was substituted for the talc as the nucleating agent in the remaining trial runs. As noted for run 635, the combination of these additions does reduce the cell size but with increasing the difficulty in making a thicker foam. Further increase of the ISTN polymer blend additive (636) resulted in a significant 12% lowering of the foam density.

Our Q1 milestone was to achieve foam density of less than 50 kg/m^3 and cell size of less than 10 micron. In the work with E-Company, we achieved significantly better density (best of 44kg/m^3) and slightly higher than desired cell size (best of 52 micron). Overall, we considered the results extremely positive, as the combination of cell size and density are the key driver in enhancing R-value and ISTN polymer blends had achieved lowering both.

Subtask 1.2 Foaming of a Polymer Blend of PS, SEBS, and Polymer-Blend-A

Significant accomplishments

A pressure vessel was successfully assembled for producing insulation foams of polymer blends with densities as low as were set by the Q2 milestones (30 ~ 40 kg/m^3). By continuing to foam new polymer blends in the vessel, we expected to further reduce the cell size, while maintaining the low foam densities, ultimately achieving better insulation value. Additionally, a factory-scale production test was successfully carried out on July 5th, 2014 at Hoswell plant in Shanghai. A new type of exfoliated clay with the potential of further reducing cell size and material costs was tested with foam extrusion of the PS and SEBS blend. Low density insulation samples (30 ~ 35 kg/m^3) were produced in factory quantity and their physical and thermal properties were evaluated. This new clay exfoliation technology had been preliminarily demonstrated in house, with significant implications in making superior thermal insulations from clay nanocomposites to be competitive on cost per R basis.

Detailed discussion

Prior to assembling the pressure vessel in Q2, all foaming experiments were done at outside locations. In order to more effectively respond to results of our foaming experiments and adjust the polymer blend recipes to further reduce foam density and cell size, we set-up a pressure vessel in house to routinely test foaming of novel polymer blends. A Parr 450 ml non-stirred vessel (N4767-O-SS) was equipped with Viton™ O-ring and quick seal closure head with a latch locking mechanism and ¼ inch ball valve for rapid pressure release. The head included gas inlet needle valve, 3000 psi rupture disc, and type J thermocouple. The split ring enclosure system allows the vessel to be opened in less than 10 seconds after depressurization (1 – 3 seconds) to aid rapid removal of the polymer foam samples. The vessel was heated with a temperature controlled jacket using the signal from the calibrated J thermocouple that was embedded inside the vessel. The vessel was pressurized with carbon dioxide (up to 2100 psig) by using a Haskel gas booster pump.

For each composition of polymer blend and additives, the vessel temperature and pressure were optimized to obtain the lowest foam density, as shown in Figure 5.

We coupled several vessel experiments with one full-scale factory foaming experiment (July 2014) to explore more thoroughly the details of using homogeneous nucleation to create submicron cells while preserving the low density. Several significant results were obtained and analyzed for further improving our strategy of reducing cell sizes at above 90% porosity.

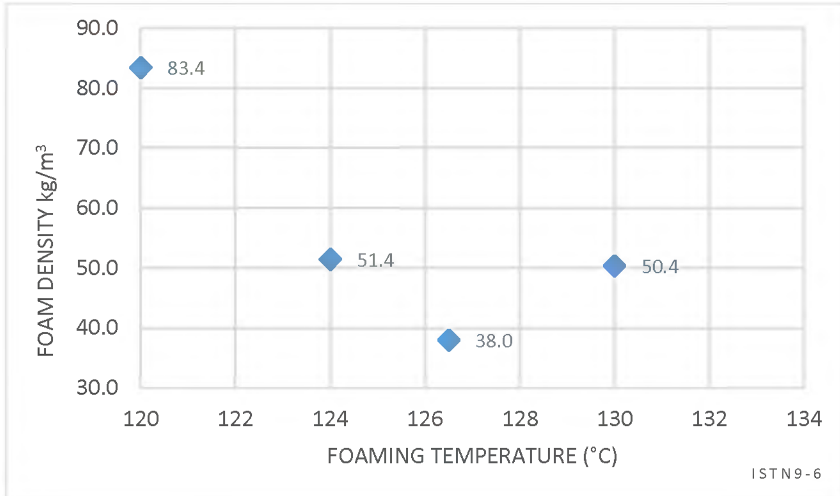


Figure 5. PS/SEBS/clay/Al foamed in the pressure vessel. Carbon dioxide pressure of 2000 psi (138 bar). Release rate of 50 bar /sec.

(1) Density reduction – In order for new insulation to be competitive on a per R basis, the foam density has to be kept below 40 kg/m^3 while reducing the cell size. In our factory test, we had tried to increase the homogeneous nucleation by elevating the level of exfoliated clay additive. The foam samples did show significantly increased nucleation but also a rise in density (from 33 to 36 kg/m^3). While this trend is in agreement with prior research observations in cell size reduction experiments, it did manifest the need of an improved strategy in our subsequent experiments and tasks in order to further reduce cell size without compromising the insulation densities.

(2) Cell size reduction – With the new pressure vessel in-house, we had the flexibility to design more new foaming experiments and blend recipes that could implement the idea of using blend domains and exfoliated clays to confine the bubble expansions and thereby prevent their coalescence. With this flexibility, we then planned in later work to add soft domains (polyethylene) in our polymer blends to promote foaming in the confined domains in a blend, and incorporate exfoliated clay additives as barriers against bubble coalescences after increasing the amount of critical nuclei in foaming. These new approaches were then implemented pressure vessel and factory extrusion experiments.

(3) Clay exfoliation and new polymer blend – As mentioned above, significant discovery in our work on the polymer blends was the full exfoliation of clay layers in our bench experiments with using a silicone-based surfactant that could also promote gas nucleation within the clay galleries. The significant potential of this innovation includes the possibility for a new platform technology in foaming superior insulations that could further drive down the material and processing costs of a key component in our new insulation recipe.

Subtask 1.3 Foaming of Polymer-Blend-A-Clay Composite

Significant accomplishments

Ingredient	% mix
PS	80.0%
Blend-A	10.0%
SEBS	9.0%
ExtenderA	0.5%
ClayB	0.5%

Table 6. ISTN-14-7.

In Q3, we began foaming polymer blend material batches for the new generation of insulation products. To recap, present building foam insulations mostly made with one polymer (polystyrene). Our objectives in foaming a blend of polymers using our technology enhancements from previously were to create superior insulation performance and improve mechanical resilience. One focus in this work was to use Polymer-Blend-A based foam insulation, which had been produced by one of our collaborators. At the start of our work, the foam density was about 60 kg/m^3. We sought to foam a blend of this polymer with SEBS to further reduce foam density to 40 kg/m^3 and thus, simultaneously reduce thermal conductivity as well as insulation cost (lower density = less solid materials, less costs). Preliminary foaming of polymer blends containing Polymer-Blend-A (example shown in Table 6) were performed in the pressure vessel and we were successful in foaming PS-Polymer-Blend-A-SEBS blends. Batches (2 kg) of dried polymers and additives were compounded in a Davis Standard twin screw extruder then injection molded into 7 mm thick plaques and then foamed in the pressure vessel to a density as low as 39.4 kg/m^3.

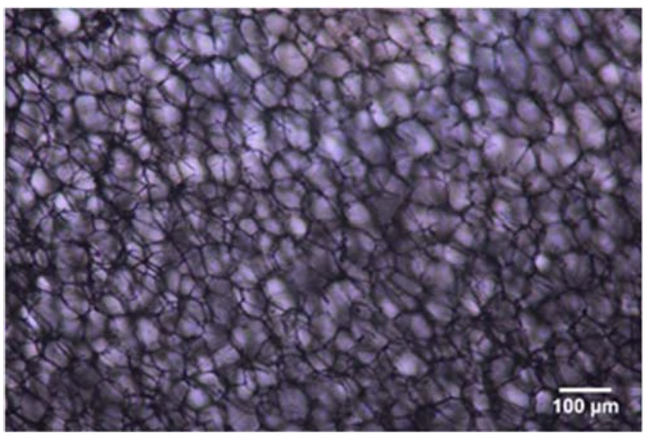


Figure 6. Fine cell structure of pressure vessel CO_2 foamed ISTN-14-7. Foam density = 39.4 kg/m^3.

Separately, we had access to the full-production foaming line at Hoswell plant for testing. We made master batches of polymer blends containing clay, SEBS, m-PE, and PS for testing production of a polymer blend insulation at the manufacturing scale. The test was successfully conducted at the Hoswell Shanghai factory and reached our low-density target of 30 kg/m^3. Factory test run data and results are shown below in the detailed discussion section.

We have successfully developed the new clay exfoliation scheme that can substantially reduce the processing cost of clay composite and be used as a platform technology for making future composite insulation material, especially for a generation of high-temperature thermal insulation material. The newly invented facile clay exfoliation technology was successfully implemented with making Polymer-Blend-A-Clay nanocomposite providing an option of blending Polymer-Blend-A in XPS through conventional low-cost foam extrusion process.

Detailed discussion

We had prepared master batches of PS and m-PE using SEBS as a compatiblizer. Clay was added into all batches as an additive to control bubble nucleation and coalescences. The master batch was mixed with variable amounts of virgin PS in the foaming extruder and the blend was extruded at a throughput of 450 kg/hour to make boards of foam insulation. The following table is a summary of the factory trial data with various compositions and run conditions. Samples are made for evaluation of mechanical and thermal properties.

Final PS	SEBS	Clay	SEBS-MA	m-PE	Density	CO_2 kg/hr.	Ethanol kg/hr.	Die P bars	Die Temp.
92.2%	0.0%	0.0%	0.9%	6.7%	31.2	12	12	80	122.5
87.2%	0.9%	0.5%	1.2%	9.7%	30.3	12	12	79	121.8
64.5%	3.6%	1.8%	3.0%	25.5%	31.3	12	12	81	120.7
78.4%	1.8%	0.9%	2.0%	16.1%	29.6	12	12	80	121.2
50.6%	5.5%	2.7%	4.1%	34.8%	36.4	12	12	95	120.3
70.0%	5.5%	2.7%	1.8%	18.2%	29.7	12	12	79	120.5

Table 7. July 2014 factory trial results.

Polymer blend (PS, m-PE, and SEBS) foams made at the production line had all achieved the densities lower than 40 kg/m^3 as shown by the data in the summary table above. The densities were gradually increased with more PE content in the blend (for example, 36.4 kg/m^3 for 35% m-PE). Some higher PE amount samples did show some shrinkage after production, a result reflecting the nature of a softer polymer. We theorized that this phenomenon could be overcome with using a different combination of blowing agents.

Subtask 1.4 Production of R-6 per inch Building Insulation using PS/POLYMER-BLEND-A/Clay Material Composites and CO_2 blowing agent

Significant accomplishments

In the polymer blend work in the first year, we achieved the following major goals towards the development of the new building insulation:

(1) Foaming of a polymer blend to small cells and 97% porosity was demonstrated on a full-scale extrusion line in a factory trial conducted in October 2014 (Shanghai).

(2) A new clay exfoliation scheme was successfully developed in our laboratory, and we have since been developing the scale-up for providing future large-scale factory trial. The new technology was a natural gateway into the Year 2 work (introducing nanotechnology to the R-6 per inch base to make super-thermal insulation values), creating the potential to minimize materials and processing costs of building insulation products in line with our project goals.

(3) A new Polymer-Blend-A based polymer blend was successfully prepared at the laboratory scale and the technology will be produced and tested at the production scale with B-Company [g] to demonstrate the improvements in a commercially applicable setting.

(4) The thermal-gravimetric-analysis (TGA) of the new Polymer-Blend-A polymer blend demonstrated a significant new application opportunity in the area of high-temperature thermal insulation (for industrial uses such as piping, as opposed to buildings). Although this application was not necessarily an expected result of the current project, we consider it an important innovation as the new technology could lead to even greater energy and environmental benefits compared to building insulation applications.

Detailed discussion

A full year of technology assessments and several large-scale factory trials of XPS insulation production provided strong indication that our goal of R-6 per inch building insulation is

g One of multiple Europe-based companies with which we collaborated on the project. Actual company name not shown in this report for confidentiality purposes.

achievable in the near-term via our use of a polymer blend material containing primarily (more than 50%) Polymer-Blend-A. Our progress toward this goal included the following:

(1) Factory trial of foaming polymer blend of PS, SEBS, metallocene PE in October 2014. – After our July 2014 factory trial, we confirmed that the goal of improving building insulation quality by 20% (to R-6 per inch) would be very difficult using PS polymer alone. Although morphology control and other die modification approaches as we had done previously were considered possibilities for improving insulation value by 20%, we believed the cost and complexity of these improvements would not make for an efficient timeline either in this project or generally, especially as due to the need for major, repeated die changes on high throughput factory production lines in realization of our previous achievement in optimizing morphology control in the CO_2 process. Considering these factors, our technical team had decided to take the route of foaming a polymer blend that contains a better insulating polymer ingredient (POLYMER-BLEND-A). Polymer-Blend-A foams have been evaluated extensively by our collaborator B-Company, demonstrating very promising results in achieving better R-value and R-value sustainability, but for both cost and production purposes was never a consideration as a commercial product. For example, foaming a polymer blend is much more difficult by nature because of problems in compounding, uniform dispersing, and subsequent foaming. However, the ISTN team successfully overcame many of these adversities to consistently produce low-density foams on a production-scale (700 kg/hour) extruder with blends of PS, SEBS, m-PE, and clay at a wide range of mixing weight ratios.

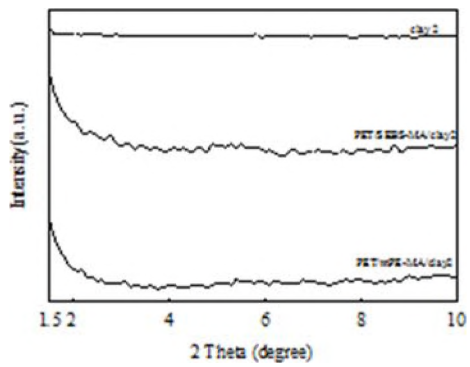


Figure 7. XRD of silicone modified clay and in batches of Polymer-Blend-A/SEBS and Polymer-Blend-A/mPE blends.

(2) Additives innovations – A major barrier of making microcellular insulation foams at affordable costs is to reduce the material cost of the special additives, in particular the cost of surfactant intercalated clays. We had achieved a major technology breakthrough by inventing a new silicone surfactant that can completely exfoliate clay (more effective than the current clay intercalation) with simple procedures. We expect this would significantly lower the material and processing costs of clay exfoliation and have been scaling up this process to produce clays for future Polymer-Blend-A blend foaming. The X-ray diffraction (XRD), shown in Figure 8, conclusively demonstrates complete clay exfoliation after treatment at pH=3.5.

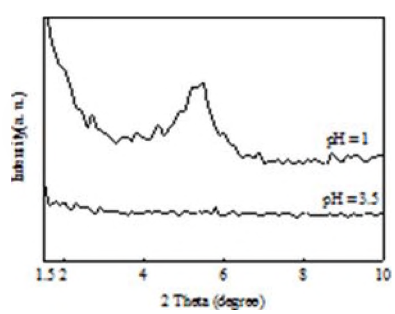


Figure 8. XRD of silicone modified clay and in batches of Polymer-Blend-A/SEBS and Polymer-Blend-A/mPE blends.

(3) Polymer-Blend-A blend at lab scale – Following the success of the low-cost clay exfoliation, we further studied the process of making a polymer blend of Polymer-Blend-A, SEBS, and treated clay to determine if such blend composite can be made uniformly with standard compounding processes. Our laboratory work accomplished a major advancement in uniformly blending a composite of Polymer-Blend-A,

SEBS (or m-PE), and clay in a ratio of 80:20:3. Again, the small angle X-ray data showed a successful blending and clay exfoliation as indicated by the diagrams in quarterly report.

(4) Polymer-Blend-A thermal properties – The most substantial discovery of this quarter was from the studies of thermal properties of the Polymer-Blend-A blends made in our laboratory. The Polymer-Blend-A blend samples made from the above compounding experiments were tested by TGA (Thermal-Gravimetric-Analysis). The data shown below demonstrated that Polymer-Blend-A thermal insulation not only have the potential of upgrading present XPS insulation by 50% with competitive cost, but also could become the base blend for a new generation of high-temperature thermal insulation with energy and environmental benefits much higher than building insulation.

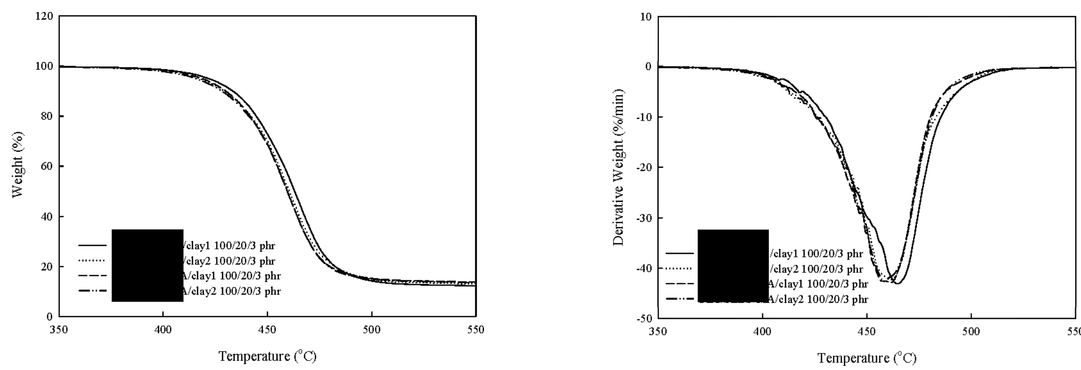


Figure 9. TGA analysis.

(5) Trial work with industrial partner specializing in Polymer-Blend-A – We established a non-binding partnership with B-Company to evaluate various foaming technologies related to Polymer-Blend-A, which is a key material in their business. The partnership includes evaluation and due diligence of technologies developed by ISTN (including in the current project), and the potential for cross-licensing of company technologies. One of the initial steps of this partnership was to schedule a production trial of several ISTN proprietary insulation recipes at B-Company facilities. This trial would allow ISTN to conduct production-scale foaming experiments at no cost, while displaying the technology for possible licensing (to B-Company) or further collaboration in commercialization. We began the first stage of the trial work in early November 2014, identifying third parties near B-Company's manufacturing plant in Europe capable of sourcing certain materials in ISTN's recipes at an appropriate scale. While Polymer-Blend-A can be sourced very simply, there was additional complexity for other additives, which required sourcing from a nearby location in the EU due to hazardous materials regulations making transport from an outside location very difficult and more time-consuming. Suppliers included Keyser & Mackay (Netherlands) and MateriaNova. B-Company funded all materials and compounding costs for their trials.

(6) R-6 per inch prototype – In the first year, we had already refined our polymer formulations and blowing processes in each factory trial such that densities went from the range of above 40 kg/m^3 to 30 kg/m^3. Meanwhile, pure Polymer-Blend-A foam board has 60 kg/m^3 density and R-value of 5.2 prior to any modifications (measurements by Oak Ridge National Laboratory). Our modeling indicated that with the higher thermal resistance afforded by Polymer-Blend-A and the initial promising results in our blend work in the lab and pressure vessel, that a full-scale production of Polymer-Blend-A-blend foam at or below density of 40 kg/m^3 could yield

R-6 per inch insulation value.

(7) 97% porosity and 30 kg/m^3 density – Through Task 1.0, we gradually improved the key insulating properties of both porosity and density of various polymer-blend insulation materials. A summary of the progress is included in the table below. As background, porosity and density are interrelated; an improvement of 96% to 97% porosity indicates a corresponding reduction in material solids, from 4% to 3%, which translates to a significant reduction in density (and materials costs) of roughly 25%.

	Q1	Q2	Q3
Blend	PS-SEBS	PS-SEBS-clay	PS-SEBS-clay
Density	44 kg/m^3	35 kg/m^3	30 kg/m^3
Porosity	95.80%	96.70%	97.20%

Table 8. Improvement in blends and key performance metrics.

(8) Manufacturing costs of < \$0.40/board-ft. – Our last production trial at Hoswell in the first year yielded insulation foam board with costs of less than \$0.40/ft^2, allowing for a commercially viable insulation product given that it is both performance- and cost-competitive to state-of-the-art XPS products on the market. Similarly for Polymer-Blend-A blends of our product, our analysis showed acceptable production costs at various R-values and densities for one of the trial's foam recipes (highest concentrations of clay, SEBS and Polymer-Blend-A to account for highest possible batch cost; most conservative estimate).

Production assumptions						
Production volume (kg)	**100,000**	Number of extruders	1.0	**Raw materials mix:**		
Foam density (kg/m^3)	32.50	x extruder hours per year	6,000		% of mix	Pricing ($ / kg)
Production volume (board-ft.)	1,303,926	Extruder capacity (hours)	6,000	CO_2	10.00%	$0.14
				Additive 1	2.00%	$10.00
Production volume (kg)	100,000	Extruder utilization (hours)	125	Additive 2	11.50%	$8.00
÷ extruder throughput (kg/hour)	800	÷ extruder annual hours	6,000	Polymer	86.50%	$2.10
Extruder utilization (hours)	125	% utilization of annual total	2.08%	Total raw materials	100.00%	$2.95

Product cost			
Raw materials:	$	$/kg	$/board-ft.
CO_2	$1,423	$0.01	$0.00
Additive 1	$20,000	$0.20	$0.02
Additive 2	$92,000	$0.92	$0.07
Polymer	$181,650	$1.82	$0.14
Total raw materials	$295,073	$2.95	$0.23
Packaging:			
Production volume (m^3)	3,077		
x Packaging cost ($/m^3)	$1.00		
Total packaging cost	$3,077	$0.03	$0.00
Shipping:			
Production volume (m3)	3,077		
x Shipping cost ($/m^3)	$25.22	79.28704	$2,800.00
Total shipping cost	$77,615	$0.78	$0.06
Total product cost	**$375,765**	**$3.76**	**$0.29**

Processing cost			
Electricity:	$	$/kg	$/board-ft.
Extruder utilization (hours)	125		
x Power consumption (kW)	350.0		
x Energy cost ($/kWh)	$0.0705		
Total electricity cost	$3,082	$0.03	$0.00
Labor:			
Number of extruders	1.0		
x Engineers per extruder	3.0		
x Salary	$100,000		
x % utilization of annual total	2.08%		
Total labor cost	$6,250	$0.06	$0.00
D&A:			
Number of extruders	1.0		
x Price per extruder	$4,000,000		
÷ useful life	10.0		
x % utilization of annual total	2.08%		
Total allocated D&A	$8,333	$0.08	$0.01
Total processing cost	**$17,666**	**$0.18**	**$0.01**
All-in production cost	**$393,431**	**$3.93**	**$0.30**

Price comparison	Existing HFC XPS	1st-Gen. CO2 XPS	2nd-Gen. CO2 P-blend
Raw materials cost ($/kg)	$3.60	$2.95	$3.55
Processing and other costs ($/kg)	$1.07	$0.98	$0.88
Foam density (kg/m^3)	30.00	32.50	37.50
Production cost ($/board-ft.)	$0.33	$0.30	$0.39
% margin	34.0%	34.0%	34.0%
Sales price ($/board-ft.)	**$0.50**	**$0.46**	**$0.59**
÷ R-vaiue per inch	5.0	5.0	7.0
Price per R-value ($/R/board-ft.)	**$0.1000**	**$0.0914**	**$0.0847**
% cushion over existing		***8.6%***	***15.3%***

Maximum allowable price ($/board-ft.)					
R-value per inch					
5.00	6.00	7.00	8.00	9.00	10.00
$0.50	$0.60	**$0.70**	$0.80	$0.90	$1.00

Sales price ($/board-ft.) sensitivity analysis					
Raw material cost ($/kg)	Foam density (kg/m3)				
	30.00	32.50	35.00	37.50	40.00
$2.95	$0.46	**$0.46**	$0.45	$0.44	$0.44
$3.00	$0.47	$0.46	$0.46	$0.45	$0.45
$3.25	$0.50	$0.49	$0.49	$0.48	$0.47
$3.55	$0.53	$0.53	$0.52	**$0.51**	$0.51
$3.75	$0.56	$0.55	$0.54	$0.54	$0.53

1st-Gen. Price per R-value ($/R/board-ft.) sensitivity analysis					
Raw material cost ($/kg)	Foam density (kg/m3)				
	30.00	32.50	35.00	37.50	40.00
$2.50	**$0.0825**	**$0.0810**	**$0.0796**	$0.0785	$0.0774
$2.75	**$0.0883**	**$0.0868**	**$0.0854**	$0.0843	$0.0833
$2.95	**$0.0930**	**$0.0914**	**$0.0901**	$0.0889	$0.0879
$3.00	$0.0941	$0.0926	$0.0912	$0.0901	$0.0891
$3.25	$0.1000	$0.0984	$0.0970	$0.0959	$0.0949

2nd-Gen. Price per R-value ($/R/board-ft.) sensitivity analysis					
Raw material cost ($/kg)	Foam density (kg/m3)				
	35.00	37.50	40.00	42.50	45.00
$3.00	**$0.0742**	**$0.0742**	**$0.0742**	$0.0742	$0.0742
$3.25	**$0.0790**	**$0.0790**	**$0.0790**	$0.0790	$0.0790
$3.55	**$0.0847**	**$0.0847**	**$0.0847**	$0.0847	$0.0847
$3.75	$0.0886	$0.0886	$0.0886	$0.0886	$0.0886
$4.00	$0.0934	$0.0934	$0.0934	$0.0934	$0.0934

Figure 10. Manufacturing cost analysis.

B. <u>Task 2.0 Secondary Nanostructure to Enhance Insulation Value</u>

Subtask 2.1 Intercalation of Chemical Foaming Agent within Clay Molecules

<u>Significant accomplishments</u>

The most significant technology advancement in this work was the verification of the benefits of the clay treated with our proprietary silicone polymer surfactant (details given in the following section) and the success in scaling up the clay intercalation technology from the laboratory to a small pilot scale. We partnered with Taiwan Surfactant Company and produced the first large batch of silicone intercalated clay in the amount of 300 grams and shipped the sample to B-Company for larger scale tests of clay exfoliation in making Polymer-Blend-A, as well as nitrile rubber insulation foams. These tests were informative for the next phase of scaling up, which would be focused on producing sufficient amounts of silicone treated clay to support larger volumes of Polymer-Blend-A foaming at B-Company and an XPS production-scale trial to be scheduled later.

A major benefit of using a polymeric silicone surfactant for clay exfoliation is its higher thermal stability. The value of our approach was further supported by the instability of other clay treated with smaller, cationic surfactants in the Polymer-Blend-A foaming trial at B-Company. Other independent studies [8] have also documented the inadequate thermal stability of the quaternary ammonium surfactants in thermoplastic processing. The TGA curve of our treated clay showed significant improvements in thermal stability as demonstrated by the comparison diagram below. Figure 11 at left displays first derivative TGA curves of commercially treated clay from BYK Additives. Figure 12 at right is the TGA of ISTN clay(2) along with data of its composite with Polymer-Blend-A, both showed much higher thermal stability by comparison).

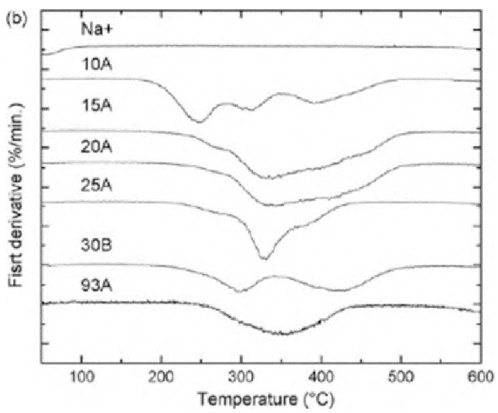


Figure 11. DTGA curves of several commercial organoclays.

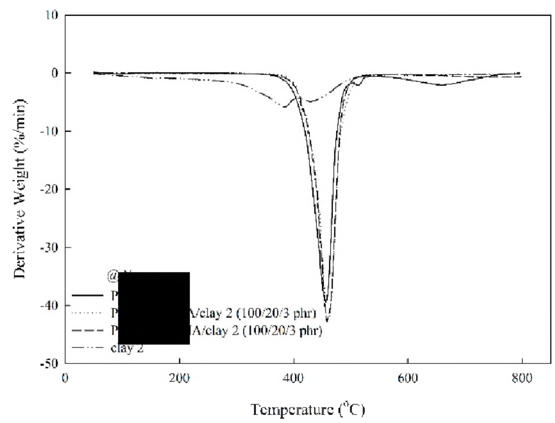


Figure 12. TGA of ISTN clay(2) along with TGA of its composite with Polymer-Blend-A.

The silicone surfactants we used to exfoliate clay were designed primarily for interaction with the acidic hydroxyl groups on clay surfaces, and can be further upgraded even for higher thermal tolerance. We had demonstrated in laboratory experiments that incorporating additional silicon block copolymers composed of both – Si–C=C– and –Si–H functional groups can father raise thermal durability by cross-linking silicone network through a hydrosilation reaction between the hydride and vinyl groups. New silicone block copolymers will be synthesized by using the same protocol of making the PDMS surfactant from two special silicone oligomers, D_4^H and D_4^V, of 2:1 ratio. The silicone oligomer structures of D_4^H and D_4^V are show at right. Preliminary studies demonstrated significant thermal durability of a silicone polymer made by crosslinking D_4^H and D_4^V in 2:1 ratio. A preliminary TGA curve indicated the new material's exceptional thermal tolerance up to 600 °C, and suggested potential to become a base material for the new generation of high-temperature thermal insulations.

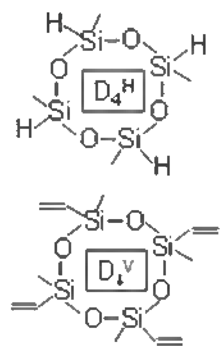


Figure 13. Cross-linked silicone network.

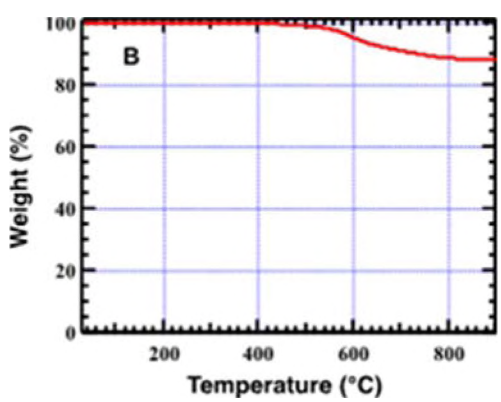


Figure 14. Clay performance over temperature increases.

<u>Detailed discussion</u>

Throughout Q5, initial foaming experiments were started with the use of hydrophilic polymers in conjunction with the incorporation of our silicone treated clay additive. PVA and starch polymers were blended with treated clay and foamed in a pressure vessel to determine whether the mixtures have the sufficient melt rheology and strength to be foamed to low densities.

Also, in February 2015, materials sourcing and compounding for recipes to be used in an additional full-scale factory production trial were completed after resolution of several logistical complexities, including the requirement of sourcing certain additives locally in the EU (hazardous materials regulations made transport from an outside location very difficult and not practical for this project's timeframe) and delays with one key additive supplier, who was extremely slow in responding from December through early January.

In March 2015, the B-Company team conducted a "non-foaming" trial of the previously compounded recipes using a Leistritz, L/D=50, twin-screw, co-rotating extruder, absent of blowing gases (e.g. CO_2) in order to predict the safety of foaming the compounded materials. An unexpected result of the non-foaming trial was that the pressure/torque for the test materials was lower than the stable foaming limit, meaning that moving directly into a foaming production trial with those materials would create a safety risk for the line operator(s). While not desirable, this result was valuable in one aspect, as it confirmed the suspected possibility for clay additives to degrade the polymers in specific conditions. We had anticipated this effect was possible because the clay selected for use in compounding (Cloisite 10A and 20A) tends to destabilize at and above operating temperatures required for Polymer-Blend-A foaming (250-300°C); however, we believe that our clay would perform much better as shown in the comparison diagrams in Figure 11 and Figure 12. Furthermore, our clay treatment protocol would allow the incorporation, or pretreatment, with polymer chain extenders which could neutralize this effect and still allow for safe foaming.

After the non-foaming trial, we used data analysis and two conference calls with B-Company to determine optimal modified recipes and techniques. Broadly, we determined two key modifications: 1) running certain Polymer-Blend-A mixtures with ISTN's additives package but now with no clay (to isolate effect of the clay) and 2) mixing the recipes with additives/clay directly into the extruder rather than compounding masterbatches beforehand, allowing less time for the clay to interact with and potentially degrade the polymers. 8 recipes were then run in a new non-foaming trial at B-Company.

The second set of results was analyzed for conclusions and then discussed in detail at an all-day technical meeting at B-Company's facilities in Muenster, Germany. Notably, we confirmed Polymer-Blend-A degradation is mainly driven by clay – the processing temperature is too high for organophilised clay and its decomposition products cause polyester chains scissions (significantly lower viscosity). Additionally, MA-modified polymers react with carboxyl end group of Polymer-Blend-A, leading to a big reduction of available carboxylic end groups, which affects branching and chain extension reactions with B-Company's reactive additives package. These conclusions support the need for a clay with significantly better temperature stability characteristics to enhance the foam insulation properties, and specifically highlight the value of the silicone-modified clay material that we have developed recently in the course of our project work. As mentioned, this novel material is known to have better thermal stability than existing nanoclays, and we provided samples of the product to B-Company for further testing in Polymer-Blend-A recipes as a commercial evaluation opportunity and also for a possible future foaming trial.

Overall, preliminary experiments in this portion of the project showed that cell size can be reduced toward 1 micron (one of our milestones), but the porosity is not high enough for an insulation product. Additional experiments to incorporate a higher amount of clay into a polyurethane (PU) resin were then designed and started. We hypothesized the foaming of PU is easier to control and to demonstrate the homogeneous nucleation phenomenon for cell size reduction. If proven successful, the new clay technology would have broad applicability to improving refrigeration and other specialty insulations.

Subtask 2.2 Foaming Nanocomposite of Clay and Block-Copolymers

Significant accomplishments

As we started this subtask, the most recent progresses shown in literature [9] demonstrated the feasibility of making full-scale R-6~7 per inch thermal insulation by a continuous foaming process. Notably, secondary nanostructures have been successfully incorporated into polyimide foam using silica nanopore aerogel created by a supercritical drying combined with subsequent foaming in a pressure vessel. Based on this work, we developed a process to integrate supercritical drying and foaming by a continuous extrusion process. Our continued progress in using silicone polymer surfactant for clay exfoliation and making polymer-clay nanocomposite led to a new provisional patent entitled "Facile clay exfoliation using silicone polymer surfactants" to be filed in 2016.

Based on these significant discoveries, we also prepared a separate, 3-year R&D strategy and plan to make a new generation of high-temperature thermal insulation for the application range of 200 - 400°C. This was not an expected outcome of the proposed project, but is a technology with significant value as the possible end product would be a new best-in-class material with the best insulating power at high temperatures (similar to aerogels) with cost-efficiency conducive to wide adoption (similar to mineral wool, unlike aerogels).

Detailed discussion

The team invented a new clay extraction process that lowers the processing cost and time, and successfully scaled up the exfoliation process to produce 0.5 kg of silicone-treated clay (applicable to 15 kg of foam product). As part of our collaboration with B-Company (commercial partner), the company tested clay samples for foaming of 1) Polymer-Blend-A and 2) NBS rubber insulation at its facilities in Germany. While clay exfoliation was successful, the surfactant interfered with the curing process in the extruder, preventing further large-scale foaming at this point.

Additionally, blends of Blends of PS, Polymer-Blend-A, SEBS with exfoliated clay were test-foamed in a pressure vessel. Low density was achieved (~40 kg/m3), but cell size not substantially reduced.

Subtask 2.3 Intertwined Secondary Nanostructure

Significant accomplishments

As we continued into Subtask 2.3 in Q 7, the two major developments were: (a) Applicability of the new clay exfoliation technology to first intercalate POLYMER-BLEND-B within galleries and then disperse POLYMER-BLEND-B into a PS blend; and (b) An integrated supercritical CO_2 processing scheme that allows formation of secondary silica nanostructure during the foam extrusion process, and thus the increasing of insulation value at competitive process. There were already batch experiments successfully demonstrating the effects of adding POLYMER-BLEND-B and nanoparticles for cell size reduction and R-value enhancements [10]. Our approach would not only realize these benefits in production, but do so cost-effectively to allow for a commercial product.

Detailed discussion

Prior to this project, many works had demonstrated that secondary nanostructure within cellular pores could enhance R-value 50-100% higher, but those values were not achievable without using the high-cost supercritical drying process in production. Based on accumulated successes of this project in the areas of clay processing and exfoliation, production-scale foaming of XPS with inorganic additives and polymer blends as well as results from references ("Porous Polyimide-Silica Composite: A New Thermal Resistant Flexible Material", Y. Fukubayashi, et. al., Mater. Res. Soc. Symp. Proc. Vol. 1645), we designed a processing scheme that utilizes the pressurized conditions in a foaming extruder to integrate the drying of secondary nanostructure with the foaming of primary pore structure.

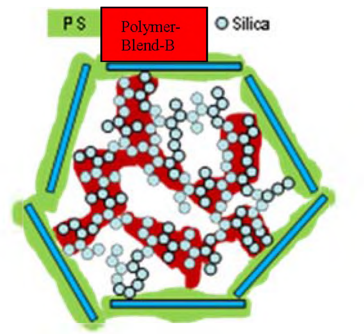


Figure 15. New polymer blend concept for super-thermal insulation value.

ISTN's design of foams with secondary nanostructure features: (1) Using exfoliated clay platelets as boundary walls to control and restrict gas expansions in foaming the primary cells in XPS insulation, (2) Nanoparticles in the form of silica alcohol-gel are to be entrapped within the clay galleries during clay's pre-exfoliation treatment for forming a nanopore structure after the alcohol is released as a foaming agent, and (3) POLYMER-BLEND-B will be blended with the clay-silica composite and be carried into the PS melt and be foamed within the clay galleries as part of the secondary structure.

PI	Silica	Decomposition	Porosity	Young's Modulus	Thermal k
85%	15%	610°C	78%	800 M Pa	0.026 W/(m·K)

Table 9. Example blend and performance characteristics.

Melt Intercalation of Modified Clay in Blends of PS and POLYMER-BLEND-B and Batch Foaming with CO_2 – A masterbatch of POLYMER-BLEND-B (Plexiglas V826, MFR =1.6 g/10 min) and MMT-ABU (7%) was prepared using a Brabender mixer at 215°C. The masterbatch was then compounded with PS resin to produce a final polymer blend with following mass composition: PS (65%), V826 (30.7%), MMT-ABU (4.3%). Pressure vessel batch foaming with carbon dioxide was conducted using soak conditions of 128°C and 2000 psi. The resultant foam was somewhat "popcorn" in morphology (Figure 1) although it did have significant areas with fine cell size (mean of 44 μm) as shown in Figure 2. Optimization of the foam blend and batch conditions will be pursued in the next period Other methods to exfoliate the clay within the POLYMER-BLEND-B by in-situ polymerization will complement the polymer blend studies The rheological properties of pure POLYMER-BLEND-B make it difficult to foam from the melt. These conditions are ideal for our previous PS blends.


Figure 16. PS/POLYMER-BLEND-B/MMT-ABU foamed in pressure vessel. Foam density ~50kg/m^3.

The innovative scheme will allow formation of nanopore structures within a continuous foam extrusion process thereby enhance the foam insulation value with affordable cost. The supercritical drying of ethanol in silica composite is utilized as part of the blowing agent to foam XPS insulation. Preliminary foaming experiments are to be conducted in a pressure vessel to optimize the material recipe and generate data for prescribe processing parameters in the design of the new extrusion process and equipment modifications. We plan to conduct a production scale foam extrusion process to assess feasibility of scaling up the new process.

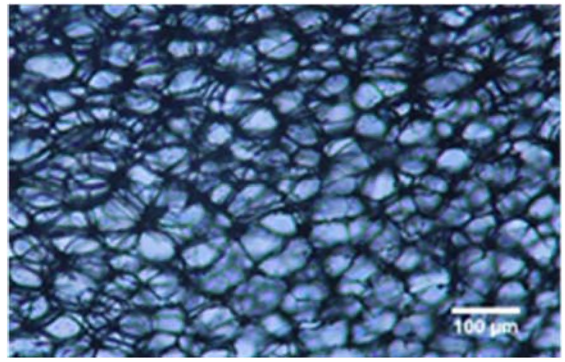


Figure 17. Optical micrograph of PS/POLYMER-BLEND-B/MMT-ABU foam. Mean foam diameter is 44μm ± 10μm.

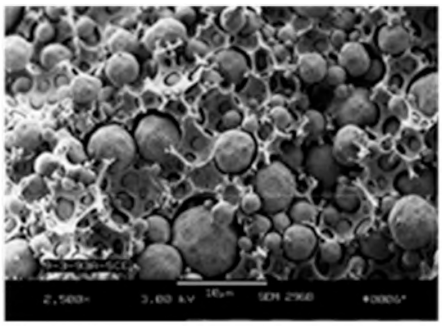


Figure 18.

Modeling calculations and our batch samples to date (Figure 16 and Figure 17) demonstrated the feasibility of R-value > 8 per inch. As the final stage of development following this project, we have planned full-scale extrusion trials focused on achieving these goals in a continuous foam extrusion production.

Subtask 2.3 Intertwined Secondary Nanostructure

Significant accomplishments

Toward the end of Year 2, we successfully completed the development of a foaming process that incorporates the drying of the secondary nanostructure with foaming a polymer blend to achieve a superior insulation (R~8 per inch) produced via cost-effective, continuous foaming process. Work in this subtask further elaborated the design of a secondary drying extruder, attached with the main foam production extruder, for compounding and delivering a polymer blend composed of clay, silica alcohol gel, POLYMER-BLEND-B under supercritical conditions into a foaming line. Carbon dioxide is to be injected into the secondary extruder and further mixed and exchanged with the alcohol in the gel; both fluids will later be utilized as the foaming gases at the die of the main foaming extruder.

Detailed discussion

Create an intertwined secondary nanostructure within larger cells of a low-density foam to further increase its R-value to that nearing a super insulation provided that the structure created does not increase foam density – Insulation value in the range of R-7~8 per inch (with all air in the cells) with foaming density less than 30kg/m^3 and full-scale manufacturing costs of < \$0.70/ft^2.

Design of foaming a secondary nanostructure

1. POLYMER-BLEND-B and silica alcohol gel are dispersed within the clay platelets and later foamed into the secondary nanostructure within a PS insulation.
2. The alcohol in the silica nanogel will be first partially adsorbed into POLYMER-BLEND-B under liquefying conditions and later be utilized to expand the POLYMER-BLEND-B polymer under foaming condition to generate the

secondary nanostructure.

Design of a side extruder to compound and deliver the blend formulation into a foaming mixture

1. POLYMER-BLEND-B and silica alcohol gel are dispersed within the clay platelets and later foamed into the secondary nanostructure within the PS insulation.
2. A side extruder is designed to compound and carry the clay-silica-POLYMER-BLEND-B batch along with alcohol and (injected) CO_2 into the main PS extruder for the formation of secondary structure and the eventual insulation foaming.

In this subtask, we successfully integrated the incremental progressions of the project such as the secondary nanostructure creation, clay exfoliation, and polymer blend foaming into an executable practice of creating nanopores by a continuous foaming process – The key innovation is to allow the fluid (ethanol) in the silica gel to be first adsorbed by a polymer blend (SEBS, POLYMER-BLEND-B) within the clay gallery, under supercritical conditions (high pressure in an extruder) so that nanopores are preserved prior and after foaming a blend. A diagram is included in Figure 15.

The continued development of this cost-competitive "super-insulation" will be pursued in our commercialization venture, for which we intend to raise venture capital funding. In this venture, our first commercial products will be R-5 to R-7 foams based on the developments in this project and the previous project.

Future technical tasks for the high R-value insulation we have been developing in this project will focus on using pressure vessel foaming of clay-silica-polymer blend to finalize designing the innovative side-extruder, and then precisely prescribe the temperature and pressure profiles in the continuous foaming production trials. This strategy will allow us to commercialize the first product within a year and gradually upgrade the technology over time to eventually develop the most technologically advanced building insulation available (before 2021, significant due to EPA regulations).

With the equipment capabilities of the full commercial venture, we will be able to scale-up the R-8 technology developed during our most recent work. While we have demonstrated that R-10 foams are possible with cost-efficient materials, we believe the best future product will be an R-8 foam that we can produce at a high throughput using the extrusion line. Foams with higher R-value than this level, regardless of materials costs, cannot be produced at a fast enough rate to make commercial sense.

C. Task 3.0 Technology-to-Market Strategy

A major focus of this project was preparing for the commercialization of a new insulation venture based on the advanced technology developed in the technical portions of this project our previous project. Our expectation was that with a portfolio of advanced insulation foam technologies, the new venture would introduce far improved product options at a reasonable cost into the U.S. and other markets, and create long overdue competition in the domestic market, which is currently dominated by two companies.

In this project, the foremost tasks in our technology-to-market strategy were in-depth market analysis, intellectual property review, identification of a commercial partner, and development of an integrated manufacturing and sales strategy to pursue in the time following the period of performance.

Subtask 3.1 Market Analysis

Market segmentation

In our analysis of the insulation market, demand for insulation materials in 2011 was $32 billion, and is expected to grow to $45 billion in 2016 (0). The two major categories of insulation products are:

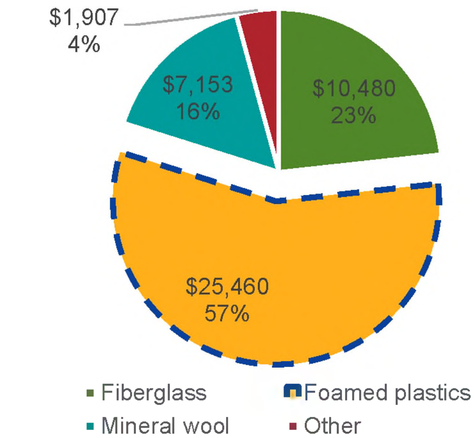
(4) Fiberglass ($10 billion market) – Fiberglass, or glass wool, is a synthetic mineral fiber made from inorganic materials including molten sand and recycled glass. Fiberglass insulation is generally made by pouring molten glass onto a spinning disc that has fine perforations in its rim. The molten glass forms fibers as it passes through the perforations. These fibers are then coated with a binder, cured in ovens, and formed into insulating batts or blankets, or chopped into loose fill insulation. Fiberglass insulation is noncombustible and does not support the growth of mildew, mold or bacteria. It also does not absorb moisture and is noncorrosive to steel, copper or other metals.

(5) Foamed polymers ($25 billion market) – Foamed polymer insulation consists of a plastic resin that is expanded through the use of a blowing agent such as carbon dioxide, hydrocarbons, hydrochlorofluorocarbons, hydrofluorocarbons, or other gases. The gases create bubbles in the foam matrix that have either open or closed cells. In open-cell foams, the ambient air occupies the voids in the foamed plastic insulation. With closed cell foams, the blowing agent is trapped within the cells, where it contributes to the thermal resistance of the product. Common resins in foam insulation include polyurethanes, polystyrenes, phenolics, polyimides, vinyl and polyethylene. One of the major foamed polymer products is XPS, which has over $5 billion in worldwide sales annually (Figure 20) representing approximately 30 million cubic meters (12.7 billion board-ft.) of volume. The major geographic markets for XPS insulation today include the U.S., the European Union, Russia and Turkey.

Market demand

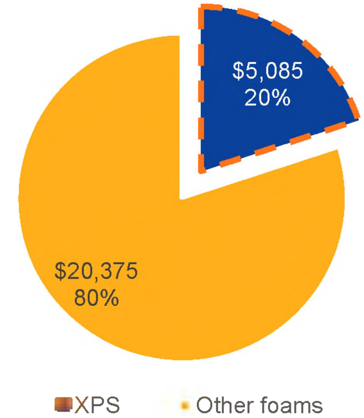
In the U.S., total insulation demand was $6.0 billion in 2011, and is expected to grow to $8.8 billion by 2016 with continued recovery in building construction activities following the recession [[11]]. Foamed polymers are $3.9 billion of the market, with XPS specifically accounting for $1.1 billion (Figure 22). On a total volume basis, XPS sales in the U.S. are 6 million cubic meters (2.6 billion board-ft.).

Dollars in millions, unless otherwise noted



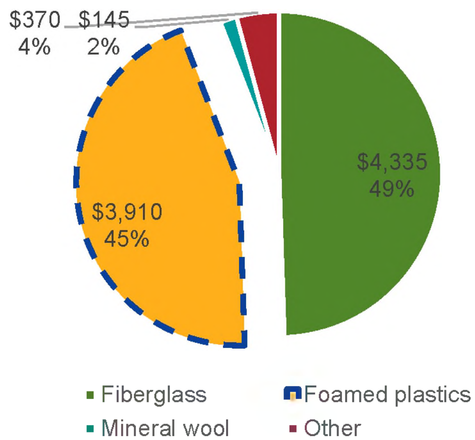
2016: $45.0 billion

Figure 19. WW insulation sales by product type.



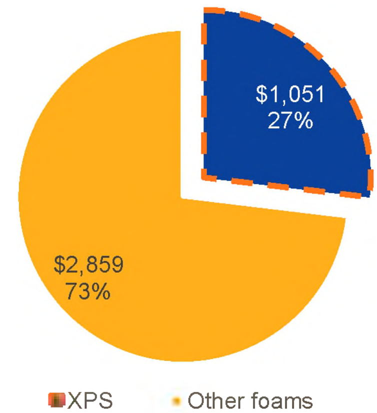
2016: $25.5 billion

Figure 20. WW annual foam insulation sales.



2016: $8.8 billion

Figure 21. U.S. insulation sales by product type.



2016: $3.9 billion

Figure 22. U.S. annual foam insulation sales.

<u>Customer segments</u>

(1) Residential construction – In 2016, projected demand for insulation materials in the residential construction market is 97 billion board-ft. of R-1 value. Applications in this market include new residential buildings and improvement/repair activities. Key drivers affecting demand in the residential market include population growth, household formation, income levels, and access to mortgage financing. For example, the 2008 economic recession caused construction expenditures to decline every year until 2011, forcing insulation sales into a correlated decline.

(2) Commercial construction – Non-residential market demand for insulation is projected at 63 billion board-ft. of R-1 value in 2016. This category includes larger, heavy-use structures such as institutional, office and retail buildings. Fixed investment at both public and private levels is one of the major drivers in commercial construction demand, and resultantly commercial insulation demand.

(3) Industrial / HVAC / other – In addition to buildings, insulation technology is essential to industrial applications such as refrigeration and high-temperature processing. Examples

include sheathing for machinery, boilers, pipes and tanks. Demand in this market will be 88 billion board-ft. of R-1 value in 2016. High-temperature insulation demand is driven by manufacturing activity, while refrigeration insulation demand depends on similar factors to the residential market, as consumers are the end user.

Product categories

A number of different insulation materials are used within the building envelope depending on their performance requirements (R-value, fire resistance, moisture sensitivity, bacteria resistance). These materials include fiberglass products such as batts and loose fill, as well as foam products such as spray foam and XPS. The most important deciding factor for purchasers is the insulation's cost per R-value. An illustration of various applications and cost-per-R-value is provided in Figure 23 below.

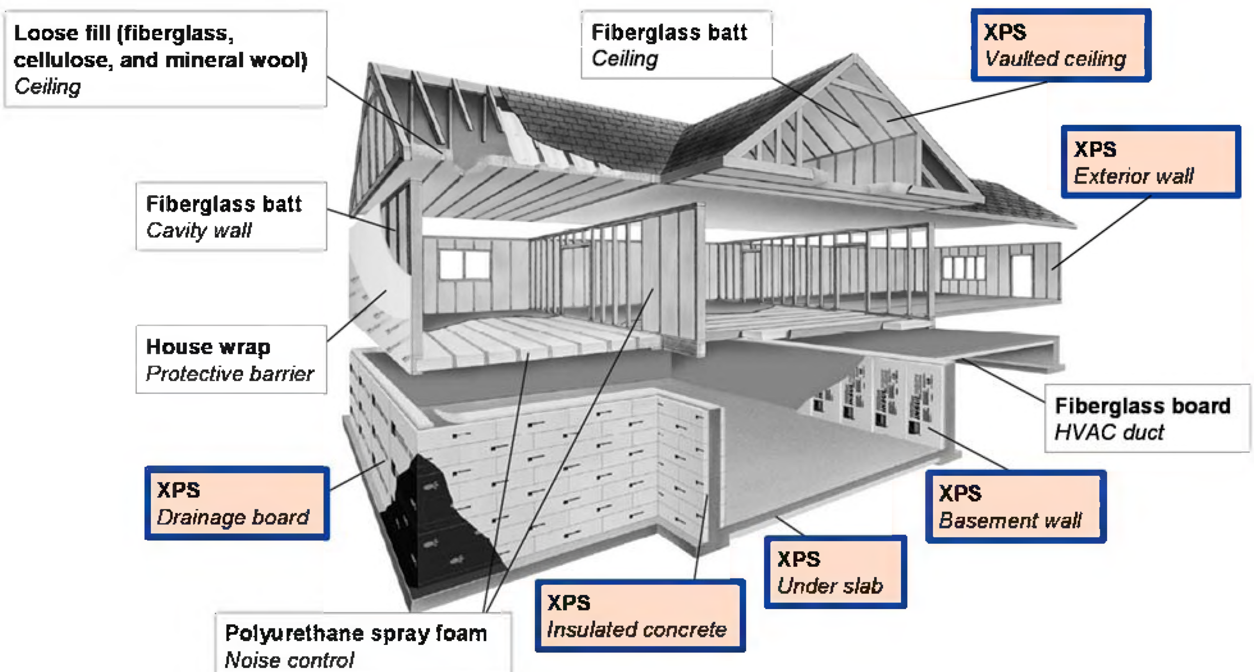


Figure 23. Diagram of applications for major insulation product types.

XPS is a versatile insulating foam used in a wide variety of residential and commercial applications, both in new construction and retrofit projects due to the material's easy installation and maintenance requirements. The core applications for XPS are described below.

(1) Residential – In below-grade (i.e., underground) foundation, XPS provides considerable thermal resistance over concrete, which itself is a poor insulator with only R-1 value for one foot of material, or the rough equivalent of a single glass pane. XPS as a foundation insulation also has the advantage of surviving well underground, where soil pressure and wet, cold conditions compromise many other materials. In exterior walls, XPS is applied as insulation sheathing (i.e., attached to the exterior framing before installing siding or other exterior covering), which adds a valuable layer of protection to the entire home from outdoor air leakage that would impair the efficiency of other insulations in wall cavity. In ceilings, attics and roofs, XPS is most valuable for use in vaulted or cathedral type ceilings with limited space (depth) for insulating the cavities between framing beams, which not only adds to the R-value of the cavity insulation, but also minimizes the impact of "thermal short-circuiting"

(accelerated heat flow) through framing studs. This is important in traditional wood frame construction, and even more so when using metal framing elements.

(2) Commercial – In commercial roofs, XPS offers good insulation value in plaza and protected membrane ("PMRA") roofing applications, in addition to extending the life of the plaza or roof by providing additional protection of the membrane from ultraviolet deterioration, weathering, physical abuse and damage. XPS is also a key component in many commercial walls, including insulated concrete sandwich panels, steel-stud framing with exterior sheathing, masonry cavity walls, and masonry wall with interior wall furring. Further, below-grade foundation is a good application, similar to residential. Lastly, building surfaces frequently require the use of waterproofing or damp proofing membranes to protect the building from moisture intrusion, and XPS is used to enhance performance over both the horizontal and vertical membrane applications.

(3) Other – Non-building uses for XPS include pipe insulation, marine use, floral and arts and crafts, utility lines, and agricultural, as well as geofoam (i.e., applied as insulation beneath highways or airport runways). The latter application particularly addresses the protection of highway infrastructure in cold-area climates where large portions of roads are covered by permafrost, which melts when trucks or heavy vehicles drive over the surface, causing the road to warp and partially sink.

<u>Competitive landscape</u>

In developed XPS markets such as the U.S. and Europe, the main insulation competitors are either large chemicals manufacturers or building materials companies with divisions focused on insulation. Major insulation companies in the U.S. market include Dow Chemical ($1.7B annual insulation sales), Owens Corning ($1.7B), Kingspan ($2.1B) and Johns Manville (<$2.5B). The largest European insulation companies include Saint-Gobain ($6.4B), Knauf ($1.5B), ROCKWOOL (<$2.6B), TechnoNICOL (<$1B), Schmid/Austrotherm (~$200M), URSA (<$585m) and PENOPLEX (<$500m). A number of smaller, specialty companies such as Aspen and Cabot produce aerogels and other niche materials, but these products are not useful in buildings due to their lack of manufacturing scalability and prohibitively high production costs. A detailed list of competitors is included in Appendix Table 10.

Of the $1.1 billion U.S. XPS market, Dow and Owens Corning together account for approximately 90%. Dow's main XPS brand is STYROFOAM™, while Owens Corning sells FOAMULAR®, PROPINK®, InsulPink® and Insul-Drain®. The remainder of the U.S. market belongs to Kingspan (GreenGuard®) and Johns Manville (AP™ Foil, CI Max®, R-Panel™).

Company	Annual revenue ($M) Total	Insulation	Description
BASF Location : Ludwigshafen, Germany Employees : 113,000 XPS brand(s) : Styrodur®	$89,934	$2,493	BASF is a multinational chemical and materials company with five operating segments: Chemicals, Performance Products, Functional Materials & Solutions, Agricultural Solutions, and Oil & Gas. In the Functional Materials & Solutions segment, the company's solutions include catalysts, battery materials, engineering plastics, polyurethane systems, automotive and industrial coatings and concrete admixtures as well as construction systems like tile adhesives and decorative paints.
Dow Chemical Location : Midland, MI Employees : 53,000 XPS brand(s) : STYROFOAM™	$58,167	$1,686	Dow is a multinational chemical and materials company with five operating segments: Agricultural Sciences, Consumer Solutions, Infrastructure Solutions, Performance Materials & Chemicals and Performance Plastics. The Infrastructure Solutions division includes Dow Building & Construction, which provides an extensive line of insulation solutions and functional ingredients that provide improved thermal performance, air sealing, weatherization and fire retardancy for construction products.
Saint-Gobain (CertainTeed in U.S.) Location : Courbevoie, France Employees : 181,742 XPS brand(s) : ISOVER	$54,553	$6,379	Saint-Gobain manufactures and distributes building materials used in the construction of housing in developed countries. The company also produces glass containers, and ceramics, high performance plastics and abrasives. The Interior Solutions segment (13% of total revenue) comprises two main businesses, insulation and gypsum, and the insulation products have three main applications: thermal management, fire protection, and sound control. Saint-Gobain is the largest supplier of insulation and related products in Europe. In the US, one in five houses utilizes the company's insulation.
The Knauf Group Location : Iphofen, Germany Employees : 26,000 XPS brand(s) : Knauf Insulation XPS	$8,151	$1,500	Knauf is one of the world's leading manufacturers of modern insulation materials, drylining systems, plasters and accessories, thermal insulation composite systems, paints, floor screed, floor systems, and construction equipment and tools. The company has 150 production facilities and sales organisations in over 60 countries. As part of the Knauf Group, Knauf Insulation products include glass mineral wool, rock mineral wool, expanded polystyrene and XPS.
Owens Corning Location : Toledo, OH Employees : 14,000 XPS brand(s) : FOAMULAR®, PROPINK®, InsulPink®, Insul-Drain®	$5,276	$1,746	Owens Corning is a leading global producer of glass fiber reinforcements and other materials for composite systems and of residential and commercial building materials. The company is the top supplier of insulation in the U.S. market with ~18% share. It manufactures and sells fiberglass insulation into residential, commercial, industrial and other markets for both thermal and acoustical applications. It also manufactures and sells glass fiber pipe insulation, energy efficient flexible duct media, bonded and granulated mineral wool insulation and foam insulation used in above- and below-grade construction applications.
ROCKWOOL Location : Hedehusene, Denmark Employees : 11,000 XPS brand(s) : None	$2,616	NA	World's leading supplier of innovative products and systems based on stone wool. Key product areas include building insulation, industrial & technical insulation for process industry, marine and offshore, acoustic ceiling systems, exterior cladding, horticultural substrate solutions, engineered fibres, and noise and vibration control.
Kingspan Location : Kingscourt, Ireland Employees : 6,600 XPS brand(s) : GreenGuard®	$2,513	$2,111	Kingspan is a global leader in high performance insulation, building fabric, and solar integrated building envelopes. Three acquisitions were completed in 2014: Dri-Design, a high end architectural façade business in the US, Pactiv Insulation, a rigid foam board producer in the US, and PAL Insulation, a Dubai based supplier of ducting insulation. Pactiv produces a comprehensive range of XPS products under the GreenGuard brand which it supplies throughout the US from its manufacturing base in Virginia.
Johns Manville Location : Denver, CO Employees : 6,855 XPS brand(s) : AP™ Foil, CI Max®, R-Panel™	$2,500	NA	Johns Manville is a producer of building insulation, roofing, roof insulation and specialty products for commercial, industrial, and residential applications. The company's insulation products include formaldehyde-free fiberglass, rigid foamed plastic and mineral wool insulation. Johns Manville is a subsidiary of Berkshire Hathaway.
TechnoNICOL Location : Moscow, Russia Employees : 6,500 XPS brand(s) : XPS TECHNOPLEX, TECHNONICOL CARBON	~$1,000	NA	TechnoNICOL is the largest Russian manufacturer and supplier of roofing and water/heat insulation materials, founded in 1992. Major products include bitumen-polymer materials, rock wool, XPS, roofing tiles, and polymer membranes. The company sells primarily to Russia, CIS, the Baltic states and Eastern Europe.
Schmid Industrie Holding Location : Waldegg/Wopfing, Austria Employees : 3,000 XPS brand(s) : Austrotherm XPS ®	~$1,000	~$200	SIH is an Austria-based holding company with subsidiaries specializing in the production of building materials. The company has around 40 production facilities in 29 countries in Europe and China. Major subsidiaries include Baumit (facade, plasters, screeds), Murexin (construction chemistry), and Austrotherm (building insulation). Austrotherm is the only Austrian producer of EPS- and XPS-insulating materials, facade elements and interior-sanitary-construction products (panels). Austrotherm sells to 10 countries and has 18 production sites.
URSA Location : Madrid, Spain Employees : 2,000 XPS brand(s) : URSA XPS	$585	NA	URSA is a leading European building insulation provider focused on glass mineral wool and XPS to insulate residential and non-residential buildings, both new and being renovated. URSA has 14 production sites in 9 countries and a commercial presence in around 40 markets in Europe, Russia, the Middle East and Northern Africa.
PENOPLEX Location : St. Petersburg, Russia Employees : 1,000 XPS brand(s) : PENOPLEX®	~$500	NA	The PENOPLEX company is a large Russian manufacturer of construction, decorative and finishing materials on polymers basis. The company began its activity in 1998 starting with launching of the Russia's first production line to manufacture heat-insulating materials made of extrusive expanded polystyrene under the PENOPLEX® trademark. The company owns eight production sites, seven of which are located in the territory of Russia: in Kirishi, Novomoskovsk, Novosibirsk, Perm, Taganrog, Cheremkovo, Khabarovsk.

Table 10. Major insulation companies in the U.S. and Europe.

Demand – Insulation demand is driven by new residential construction, remodeling and repair activity, commercial and industrial construction activity, increasingly stringent building codes and the growing need for energy efficiency. Sales patterns typically follow seasonal home improvement, remodeling and renovation and new construction industry patterns, with demand tending to lag new residential construction patterns by three months. Peak season for home construction and remodeling in the U.S. generally corresponds with the second and third quarters.

Costs – The cost of insulation materials is heavily dependent on raw materials prices. For example, XPS solid content is ~85% polystyrene, so the product cost closely follows fluctuations in the commodity pricing of polystyrene resin. PS prices are driven by availability of feedstock petrochemicals and demand for numerous derivative consumer products (e.g., STYROFOAM™ cups). Separately, shipping costs are a significant portion of total insulation product cost due to the extremely light weight of a good insulation. The inability to ship extended distances (i.e., more than one day by truck) dictates the need for strategic dispersion of production facilities to maximize distribution reach and cost-efficiency.

Innovation – The predominant technologies and manufacturing methods within the insulation industry are extremely outdated. Fiberglass and foam mass production date back to the 1940s, and current processes have improved only marginally. Also, major insulation manufacturers have been resistant to innovation until facing regulatory changes. Foam insulation specifically has also faced the major technical hurdle of exceeding R-5/inch using a cost-effective production method.

U.S. regulatory change

Toward the end of the project, one major positive change was further progress toward favorable regulatory change for insulations based on clean blowing agents. On July 2, 2015, the EPA finalized a rule under the authority of the Clean Air Act and EPA's Significant New Alternatives Policy ("SNAP") Program to phase out usage of climate-damaging HFCs in several industrial applications [12]. Specific to insulation and XPS production, the agency *banned the following blowing agents as of January 1, 2021 [h]: HFC-134a, HFC-245fa, HFC-365mfc, and blends thereof; Formacel TI, Formacel B, and Formacel Z-6*. Thus, starting in 2021 all of the HFC blowing agents that are critical to current XPS will not be allowed in manufacturing. Products manufactured prior to 2021 may still be sold, imported, exported and used at any time, although any such strategies have practical limitations. For example, while a manufacturer could front-load production with HFCs prior to 2021, existing line capacity and inventory costs would limit the amount. In addition, although the rule does not prevent importation of HFC-blown XPS from other countries, the costs are prohibitively high as shipping is a significant factor in insulation cost.

Dow, Owens Corning and other key players in the XPS industry successfully lobbied for a delay of the effective date from January 1, 2017 by arguing that production line modification, third party testing and lack of energy efficient alternatives would delay the changeover. However, the major issues for these companies in reality are the lack of viable next-generation technology based on allowable blowing agents, and an unwillingness to invest in technology development and change.

Among the EPA-approved substitute blowing agents, the most reasonable HFC replacements for XPS production are Hydrofluoroolefins [i] ("HFOs") and CO_2. HFOs provide good R-values in

h Military or space- and aeronautics-related applications have one additional year to comply.

i Hydrofluoroolefins are the latest in a succession of blowing agents that started with CFC11, a chlorofluorocarbon developed in the 1960s and long since phased out because of its damage to ozone. Manufacturers later developed hydrochlorofluorocarbons ("HCFCs") in the 1990s and then hydrofluorocarbons ("HFCs") in the early 2000s as they attempted to come up with blowing agents that had a zero ozone depletion potential ("ODP") as well as a low global warming potential ("GWP"). HFOs can be considered "fourth generation" blowing agents that have both of these beneficial characteristics.

XPS, but are too expensive to meet the low-cost needs of the industry. Further, they are a proprietary technology (Honeywell) made in only one location, presenting a major risk of insufficient supply or lack of competition. In contrast, CO_2 is both abundant and extremely low cost, but has proven challenging for manufacturers to produce competitive R-value foams, a concern that Owens Corning most notably raised to EPA. ISTN's technology is unique as a CO_2-blown XPS foam that meets the thermal resistance and cost-efficiency of HFC-blown products, and is ideally positioned with a head start to capitalize on the forthcoming regulatory change.

Blowing agent	GWP	U.S. status	Cost ($/lb.)	Flammability	Foamed R-value	
CO_2	1	Acceptable	< $0.10	Non-flammable	ISTN - Best	<--- ISTN process
Exxsol Blowing Agents (HC)	5	Acceptable	< $0.50	Highly flammable	Poor	
Ecomate™ (methyl formate)	5	Acceptable	$3.00	Flammable	Poor	
HFO-1234ze	6	Acceptable	$60.00	Low flammability	Best	
Saturated Light Hydrocarbons (C3-C6)	3 to 10	Acceptable	< $0.50	Highly Flammable	Poor	
DME (dimethyl ether)	1	Acceptable	$0.50	Flammable	Poor	
HFC-152a	122	Banned as of 01/01/21		Flammable	Poor	
HFC-365mfc	782	Banned as of 01/01/21		Flammable	Good	
HFC-245fa	1020	Banned as of 01/01/21		Non-flammable	Good	
Formacel® Z-6	370-1290	Banned as of 01/01/21		Flammable	No data	
HFC-134a	1320	Banned as of 01/01/21	$4.00	Non-flammable	Best	<--- Industry standard
Formacel B	140-1500	Banned as of 01/01/21		Flammable	No data	
Formacel® TI	1330-1500	Banned as of 01/01/21		Flammable	No data	

Table 11. Analysis of all XPS blowing agents.

<u>International markets</u>

In November, 2015, the 197 Parties to the Montreal Protocol agreed to begin work on an amendment to be completed in 2016 that will phase out the global production and consumption of HFCs. First originating in 1987, the Montreal Protocol is an international treaty designed to protect the ozone layer by eliminating the production of numerous substances that are responsible for ozone depletion, such as CFCs. While details of the HFC phase-out and timing remain to be determined, the shift internationally toward replacing these gases with more environmentally friendly alternatives is a positive development for our insulation technology. As is our expectation in the U.S., the ability to introduce clean foaming technologies ahead of competitors in international markets of interest will be advantageous, especially once HFCs are banned.

Additional considerations in these markets are further discussed below:

(1) <u>Canada</u> – In Canada, annual demand for foam insulations is nearly $300 million. Foam is the highest growth segment in the Canadian insulation market, while fiberglass is the largest. The growth profile of foam and several cold-climate areas in the country are appealing for XPS and similar foam extrusion products that we intend to manufacture. Additionally, most Canadian manufacturers are small- to mid-size companies that producers a particular type of insulation material. There is a strong presence of U.S. companies that maintain production facilities in the country. Examples include such as Owens Corning and Kingspan, the latter of which makes XPS in Caledon, Ontario and Langley, British Columbia. Canada is a good opportunity for near-term expansion of our business once are able to add a facility closer to the northern U.S.

(2) <u>Middle East</u> – Historically, many buildings in the Middle East have not used insulation of any kind due to the low costs of burning domestic oil to generate electricity for HVAC. For example, in Saudi Arabia, an estimated 70% of buildings are uninsulated. However, in such hotter climates, better insulation is essential, having the ability to reduce the significant air conditioning use by approximately 40%. Recently, various forms of regulation have increased

as Gulf region countries tighten their energy efficiency policy. Examples include Saudi Electric Company's requirement that *all* new construction have insulation and be interconnected to grid, as well as numerous regulations in building code in UAE and Qatar, particularly relating to qualification for "green" designations. This market would be ideal for our insulation, particularly as we move into further generations of technology with polymer blends that have higher deformation temperature than PS to optimally perform in the desert climate.

(3) China – The Chinese economy's energy intensity (24,708 BTU per GDP$) is 3.5x that of the U.S. (7,328) and 5.5x Germany (4,457) and Japan (4,574). Insulation materials for buildings would alleviate this issue since the Chinese insulation market is underdeveloped. Past concerns with several severe fires in high-rise buildings and XPS flammability have prompted a short-term ban of XPS use in populated areas. Additionally, the installation of building insulation in China differs substantially from the U.S.; insulation foams are normally applied directly onto concrete walls as a heat and moisture barrier, instead of on the wood frame as part of the load-bearing structure. Thus, the Chinese market desires a new product substantially different from XPS in flexibility, flammability, and other improvements, for which our next generation of polymer blend technology could serve a significant need. Marketing efforts will be very important for product acceptance among Chinese architects and contractors, insulation suppliers, and importantly government regulators. Dr. Yang can employ his knowledge of insulation materials and the Chinese market, as well as his network of contacts in Taiwan and China to facilitate promotion of the new products and the market opportunity.

(4) India – Similar to China, India's mix of large population, energy waste and developing use of insulation offers tremendous opportunities for new insulation technology. While the country's energy intensity (17,486 BTU per GDP$) is better than China's today, energy consumption will likely grow at an even faster pace due to lagging development in certain population-dense areas, coupled with great demand for better living conditions. In a major development cycle for which the Indian government plans rapid infrastructure expansion, enhancing the application of thermal insulation in buildings and factories will accomplish the most significant energy savings, simultaneously raising both the standard of living and the competitiveness of the country's industrial economy. Energy savings from insulating buildings and industrial facilities could immediately relieve the burgeoning energy demands of the economy. Also, building envelope insulation will not only prolong the service lives of all HVAC equipment, it will also lower HVAC electricity costs for the general population, creating economic stimulus as a result. The hot and humid weather in many major regions of India make the insulation application arguably more attractive than in even the U.S. and China.

Subtask 3.2 Commercial Partner

In Q2, we developed a list of 140+ potential investors/partners based on existing relationships and our research of the insulation market. This list served as the starting point for our outreach to industrial companies and venture capital firms with interest in partnering on a new commercial insulation venture. Notably, we spent considerable time meeting with B-Company and their management team to discuss the opportunity of either a joint venture or other funding arrangement. B-Company generates over $500M in annual sales and produces a variety of foam insulation products outside of the building sector, making them a good fit for protecting confidentiality of our IP (a concern of working with other building insulation companies). During Year 1, we agreed on an informal basis to perform certain foaming trials at B-Company facilities in Europe, which was resource-efficient for us, strengthened the potential of a formal business collaboration, and provided certain R&D expertise to the B-Company team.

In Q4, we executed a two-way NDA with B-Company, and agreed on their funding of our production-scale foaming trials taking place in their manufacturing facilities in Europe for Task 1. Before considering the future commercial potential of the partnership, access to the manufacturing facility was of significant value to ISTN given the limited availability of similar facilities in the US. Most, if not all, such domestic facilities are owned by building insulation manufacturing competitors. Moreover, having access to B-Company's expertise in Polymer-Blend-A materials was extremely helpful in the phases of our project involving Polymer-Blend-A blend foams. Also, the company's willingness to pay for materials and compounding related to foaming trials made our work highly cost-efficient within the constraints of the project budget.

Despite clear benefits of performing our trial work with B-Company, we decided after further evaluation in Year 2 that ISTN should first pursue our U.S. commercial venture independently before engaging an industrial partner long-term. With the significance of the domestic market opportunity and our strong technology portfolio, the business opportunity is quite significant. The ideal U.S. partners would have experience in building insulation manufacturing and sales, pointing to Dow and Owens Corning; however, such companies pose a competitive risk for any partnership given the information we would be sharing. Additionally, our knowledge of the U.S. market is extensive, and limits the need for a partner in this specific market. Our goal here is to raise capital and manufacture product ourselves, while in future international markets we could partner with B-Company in Europe, and then similar companies in countries where they have the domain expertise to complement our technology.

Subtask 3.3 Intellectual Property

In Year 1, we performed detailed analysis of the intellectual property landscape for foam insulation. A summary of all relevant IP is included below, along with a comparison of ISTN's IP position as of the project end date. In our review, we ensured that none of our current or pending IP was encumbered by previous proprietary work in the field, and further that manufacturing know-how and trade secrets in our processes do not infringe on other methods.

Patent Number	Title	Priority date	Status	Company
Previous patents:				
1) US20120149793 A1	MONOMODAL EXTRUDED POLYSTYRENE FOAM	08/28/09	Abandoned	Dow Chemical
2) US8557884 B2	To enhance thermal insulation of polymeric foam by reducing cell anisotropic ratio and the method for production thereof	05/31/02	Grant	Owens Corning
3) US8568632 B2	Method of forming thermoplastic foams using nano-particles to control cell morphology	11/26/03	Grant	Owens Corning
4) US6268046 B1	Process for producing extruded foam products having polystyrene blends with high levels of CO2 as a blowing agent	10/21/99	Grant	Owens Corning
5) US7605188 B2	Polymer foams containing multi-functional layered nano-graphite	12/31/04	Grant	Owens Corning
6) US6759446 B2	Polymer nanocomposite foams	05/02/02	Grant	Ohio State University
7) US8568633 B2	Elastic particle foam based on polyolefin/styrene polymer mixtures	04/11/07	Grant	BASF
ISTN patents:				
1) US8785509	**Superinsulation with nanopores**	**05/02/08**	**Grant**	**ISTN**
2) US 14/336,393 (divisional application)	**Superinsulation with nanopores**	**05/02/08**	**Pending**	**ISTN**
3) US 61/813,390 (application)	**Advanced thermal insulation by pore morphology control in foaming**	**03/13/13**	**Pending**	**ISTN**
4) PCT/US2014/034663 (PCT application)	**Advanced thermal insulation by pore morphology control in foaming**	**04/18/13**	**Pending**	**ISTN**
5) TBD	**Facile clay exfoliation using silicone polymer surfactants**	**2015**	**To be filed**	**ISTN**

Table 12. Intellectual property review.

Subtask 3.4 Manufacturing and Sales Strategy

In Q6, we submitted a business plan detailing our manufacturing and sales strategy for the new insulation venture. The plan was based on our experience in manufacturing trials with large-scale producers of foam, and our research of the market, supply chain and other factors. The key aspects of the strategy are:

(1) Plant – The facility must be centrally located relative to large, cold-climate markets (New York, Midwest, New England, and Canada), which will limit shipping costs. As XPS insulation is a low density material, efficient shipping options (rail and highway) that minimize cost are critical in selling to customers. The plant must have a height of at least 27 feet to allow clearance for the tallest parts of the extrusion line. Estimated square footage is 60,000 $ft.^2$, which comfortably holds one line (25,833 $ft.^2$ floor space), product inventory and other company operations, and sufficient space for the future addition of a second extrusion line when increased production capacity becomes necessary.

(2) XPS board extrusion line – Core manufacturing activities will require the purchase of an XPS board extrusion line. We expect one line to produce approximately 50 million board feet (2% of the U.S. XPS market) of foam in a year, and require 3-4 technicians to operate. The equipment supplier will be Sunwell Global Ltd. a large Taiwan-based manufacturer of extrusion equipment and foaming technologies. As we have previously used Sunwell equipment on a number of our production trials of the XPS technology, maintaining this continuity ensures the scale-up of new production has similarly successful results to our previous trials. Sunwell has provided a price quotation for the extrusion line. As part of the

equipment purchase, Sunwell will provide installation services including two technicians over two weeks, an additional two technicians for two weeks of start-up and process fine tuning, and one skilled process consultant for a week of process training.

(3) Raw materials – General purpose polystyrene ("GPPS") is the major component of our first-generation XPS, comprising ~85% of raw materials. The supplier for previous factory trials was Chi Mei Corporation. For suppliers of our commercial production, we will consider Chi Mei, and have also inquired of high volume pricing from the major suppliers in the U.S. and abroad (e.g., BASF Group, INEOS Nova, Trinseo, etc.). Many of these suppliers also sell the other polymers in our next-generation blend recipes. The importance of production cost stresses the need to identify the lowest cost supplier, which may either be domestic due to minimal shipping costs, or overseas from regions where currency weakness relative to the dollar could outweigh any increased shipping costs. We also plan to commit to purchasing a reasonably large amount of GPPS upfront in order to obtain volume discounts, which is made possible by our high production volume. Blowing agents will all be sourced in the U.S., as CO_2 is extremely cheap.

(4) Product specifications – The 1st-Gen. XPS material will have R-5/inch, density of less than 40kg/m^3, and a production cost of \$0.30/board-ft. Board thicknesses will include 1-inch and 2-inch varieties. Reference values for secondary specifications are listed in Table 13. Additional consideration will also be given to the foam coloring (currently gray) and printed designs on the board, which can both be employed as branding elements.

Property	Unit of measurement	Test method	Value
Service temperature	maximum °F (°C)	N/A	165 (74)
Compressive strength	minimum psi (kPa)	ASTM D1621	15 (103)
Flexural strength	minimum psi (kPa)	ASTM C203	60 (414)
Water absorption	maximum % by volume	ASTM C272	0.1
Water vapor permeance	maximum perm (ng/Pa•s•m^2)	ASTM E96	1.5 (86)
Dimensional stability	maximum % linear change	ASTM D2126	2
Flame spread	index	ASTM E84	5
Smoke developed	index	ASTM E84	45-175
Oxygen index	minimum % by volume	ASTM D2863	24
Linear coefficient of thermal expansion	in/in/ºF (m/m/°C)	ASTM E228	3.5×10^{-5} (6.3×10^{-5})

Table 13. XPS product specifications. Reference values based on FOAMULAR product specifications.

(5) Production – Once the extrusion line is installed, we will perform pilot foaming tests of the 1st-Gen XPS product using CO_2 as the blowing agent. HFC and HFO varieties will be also produced in the pilot tests for additional benchmarking. The XPS lots produced will be tested and then refined via process and recipe optimization before conversion of pilot testing to line production. Assuming full capacity production of 6,000 hours in a year at a rate of 600kg/hour, the extrusion line will be capable of producing over 4 million board-ft. per month. This production volume will be divided among the varying board thicknesses based on customer demand.

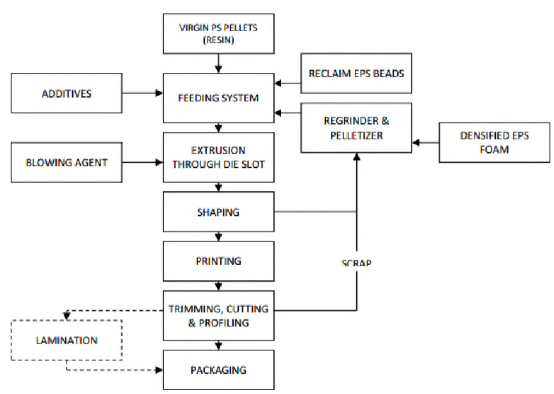


Figure 24. XPS process diagram.

(6) Quality control and certifications – Quality assurance and quality control proof is necessary in order to sell the final product. A number of product certifications will be needed for properties of the foam board, including mechanical strength, fire resistance and moisture resistance. A list of testing standards for which we will submit our product is listed below in Table 14, grouped by importance to marketability. Through our DOE project, we have also maintained contact with Oak Ridge National Laboratory ("ORNL") and National Renewable Energy Laboratory ("NREL"). We will use both groups to assist in testing our new products, with ORNL specializing in material properties such as the thermal resistance, and NREL having greater capability in studying installed energy efficiency impact of the material.

Standard	Name	Category
Core tests:		
ASTM C518	Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus	Thermal/physical
ASTM C578	Standard Specification for Rigid, Cellular Polystyrene Thermal Insulation	Thermal/physical
ASTM E84	Surface Burning Characteristics of Building Materials	Flammability
ASTM E96	Water Vapor Transmission of Materials	Moisture
ASTM C272	Water Absorption of Core Materials for Sandwich Constructions	Moisture
ASTM D1621	Compressive Properties of Rigid Cellular Plastics	Thermal/physical

Table 14. Product testing and certifications.

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