



Advanced Bus and Truck Radial Materials for Fuel Efficiency

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PPG
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Project ID: VS163



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Overview

Timeline

- **Start: 10/01/2014**
- **End: 03/31/2018**
- **BP1 completed**
 - On March 31st 2016

Budget

- **Total project funding**
 - \$1,253,269 (Total), \$939,950 (DOE)
- **BP1 Funding**
 - \$384,131 (Total), \$288,098 (DOE)
 - \$277,854 (Spent), \$208,390 (DOE spent)

Barriers

- **Technical Target**
 - 4-6% improved fuel efficiency of truck and bus radial tires
- **Technical Barriers**
 - Dispersion of fillers in natural rubber-based compounds
 - Natural rubber compound optimized for silica filler

Partner

- **Bridgestone Americas Tire Operations**

Relevance

- **Project goal: develop a novel surface-modified silica technology and demonstrate 4-6% improved fuel efficiency of truck and bus radial (TBR) tires built from the technology.**
 - Maintaining or improving tear strength and treadwear over the state of the art carbon black-filled natural rubber-based TBR tread compound
- **Past year goal (BP 1)**
 - ✓ Understand how different silica surface chemistries and surface areas are linked to dispersion performance in different rubber phases including both natural and synthetic rubbers
 - ✓ Selection of silica chemistries and surface areas that deliver the most consistent dispersions in various rubbers
 - ✓ Identify at least 1 treated silica technology that demonstrates a 10% increase in dispersion in multiple rubber compounds compared to incumbent carbon black or untreated silica technologies.
- BP 2: Develop a new TBR compound containing surface modified silica, that reduces the rolling resistance by at least 60% in laboratory testing
- BP 3: Optimize the TBR compound performance and select the final rubber compound formulations for the tire builds that will be delivered to DOE for independent testing


Milestones

Milestone	Plan * Updated - Actual Date	Status
0.1 Subcontract Executed	11/30/2014 – 12/11/2014	Completed
1.2.1 Microdispersion Technique Determined	1/30/2015 – 3/18/2015	Completed
1.2.2 Structure Determined	6/30/2015 – 10/09/2015	Completed
2.2.1 Surface Energy Effect Determined	9/30/2015 – 1/11/2016	Completed
3.1 Tread Compound Determined	12/30/2015 * 5/18/2016	Started
3.2 Tread Compound Formulas Selected	3/31/2016 * 8/30/2016	
4.2.1 Experimental Trailer Tires Produced	4/15/2016 * 12/16/2016	
5.1 Performance Gaps Identified	9/30/2016 * 3/30/2017	
6.1 Tread Compound Formula Identified	12/31/2016 * 7/03/2017	
6.2 Exp. Tread Compound Formula Selected	3/31/2017 * 10/02/2017	
7.2.1 Experimental Tires Produced	4/15/2017 * 10/20/2017	
8.1 Selection of Tire Technology for Testing	9/30/2017 * 03/30/2018	
Final Report	12/30/2017 * 6/30/2018	

Go/No-Go Budget Period 1

- ✓ Identify at least 1 treated silica technology that demonstrates a 10% increase in dispersion in multiple rubber compounds as measured by Dispergrader compared to incumbent carbon black or untreated silica technologies.

Approach

- In passenger tires silica provides fuel-efficiency gains over carbon black.
 - TBR tires predominantly comprised of natural rubber (NR)
 - NR provides the chip and tear resistance essential for TBR applications
 - NR contaminants (proteins, metal ions, etc.) are believed to interfere with the *in situ* coupling reaction required to effectively disperse silica in NR, yielding poor filler dispersion, tire performance, and processing
 - Silicas no longer provide the same benefits
- 
- PPG investigated the ability of surface treated silica to overcome the NR contaminant problem and observed good dispersion in NR and provided RR improvements over carbon black

“Innovating the silica surface for Improved NR truck tire vulcanisates” *Tire Technology International* 2/2014.

“Functionalized silicas for improved NR truck tire vulcanizates” *Rubber World* (2014) 249(2), 19-24.

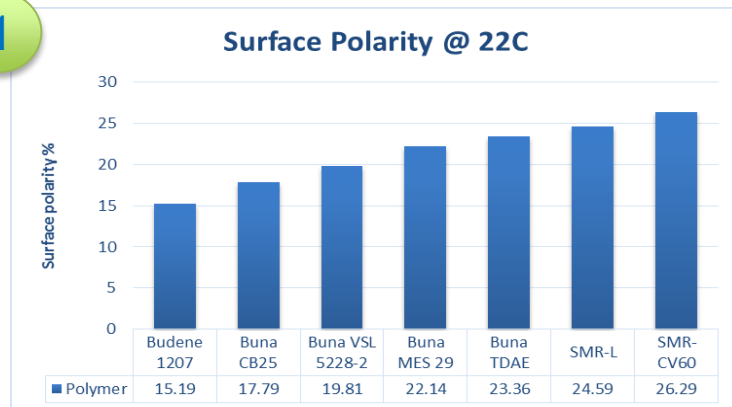
“Bringing Innovation to the Surface: Functionalized Silicas for Improved Natural Rubber Truck Tire Vulcanizates,” 184th Technical Meeting of the ACS Rubber Division, October 2013, #33.

“Agilon Performance Silicas in Natural Rubber Truck Tire Tread Compounds” 180th Technical Meeting of the ACS Rubber Division, October 2011, #70.

Strategy & Summary of Past Work

Identify Interfacial Behavior of Key Polymers

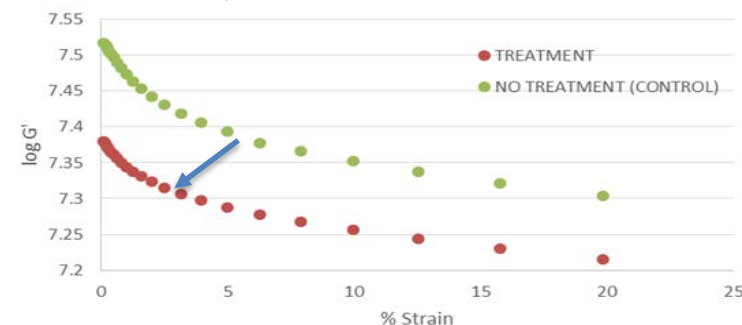
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Test Silicas in Single Polymer Model Formulations

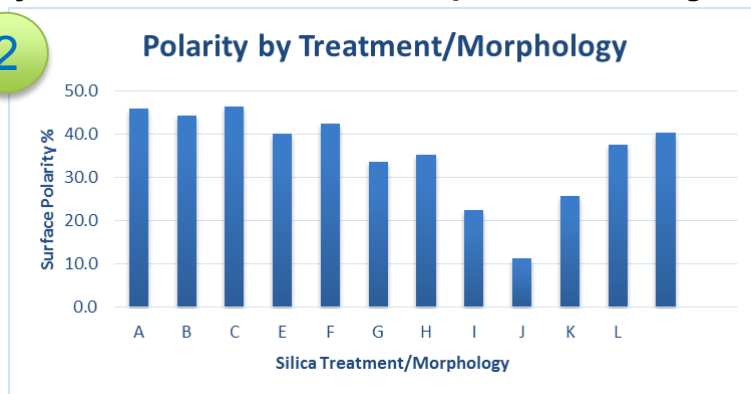
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Reduced Payne Effect, Improved Dispersion



Identify Silica Chemistries for Improved Wetting

2



Polymer Blends for Optimized Wear, Tear, RR and Safety

4



Select modifiers/morphologies that offer:

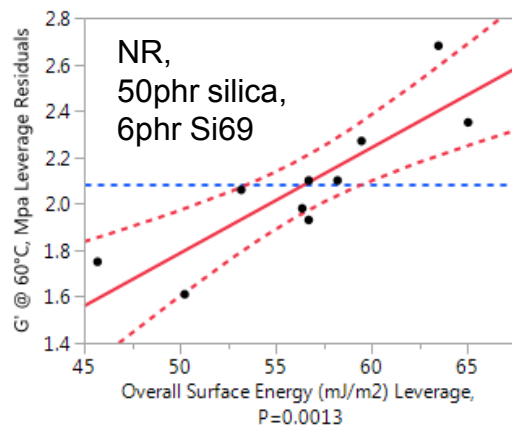
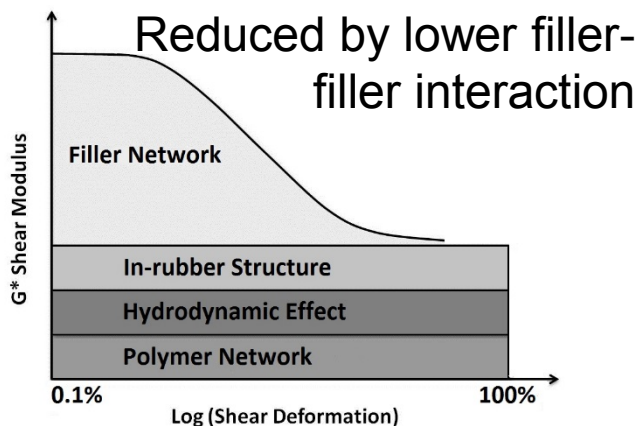
- Equal or better tread wear, tear strength
- 60% lower RR than carbon black control

Goal: Optimized Tread Compound/Tires

Technical Accomplishments and Progress

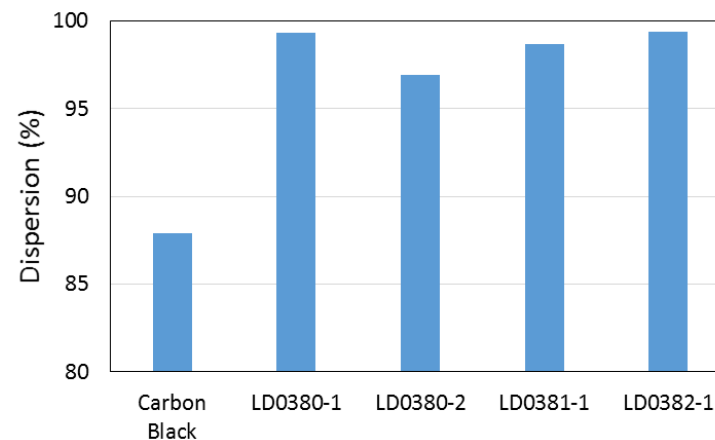
Improved Filler Dispersion in NR Compound

Payne effect



Dispersion by Dispergrader

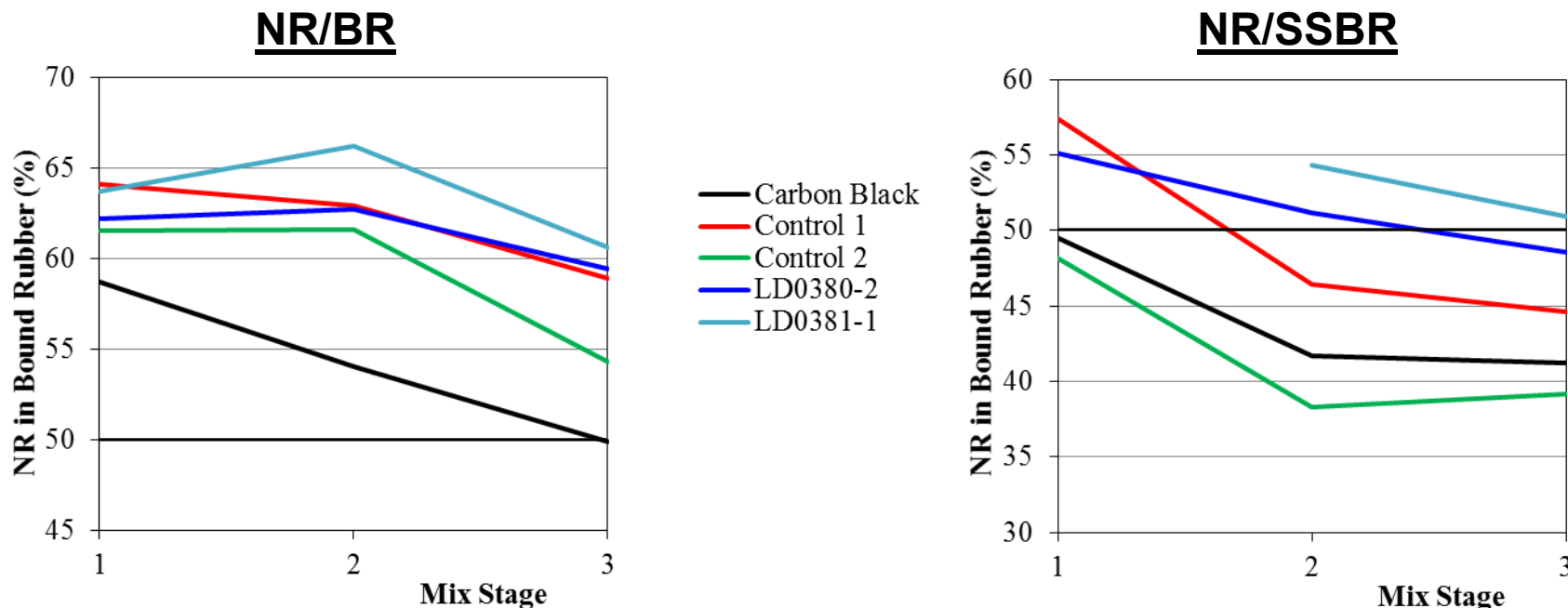
- Silica prototypes with low surface energy selected to move forward



- Lower silica surface energy shows reduced Payne effect
- Improved dispersion confirmed by Dispergrader

Filler Distribution in Polymer Blends

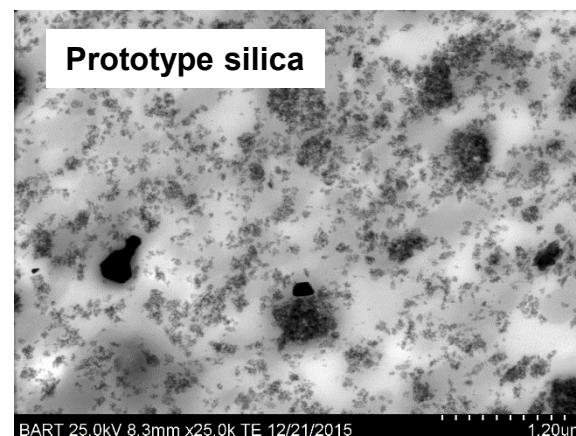
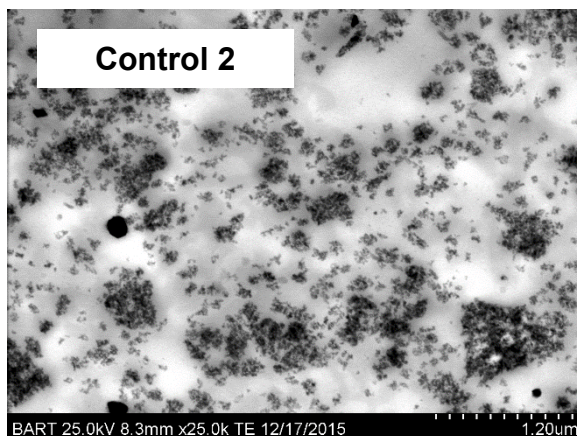
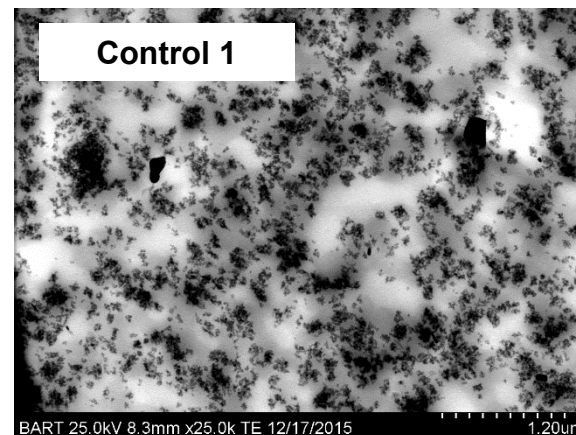
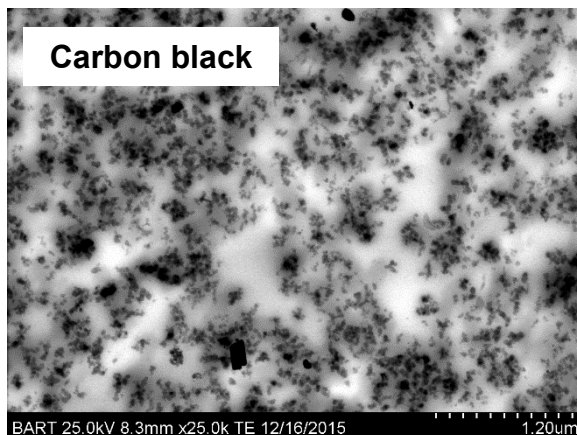
- Compounds with 50/50 NR/synthetic rubber, 60phr filler
- Unbound rubber extracted after each mixing step
- ^1H NMR analysis performed to determine composition of rubber bound to filler



- Fillers incorporate NR initially and then shift towards synthetic rubber
- Carbon black preference: $\text{SBR} > \text{BR} \approx \text{NR}$
- Control silicas preference: $\text{SBR} > \text{NR} > \text{BR}$
- Silica prototypes preference: $\text{SBR} = \text{NR} > \text{BR}$

Polymer Blends - SEM Microscopy Results

Dispersion in NR/SBR Polymer Blends



SBR: dark phase; NR: white phase

- Microscopy data agrees with NMR.
- Prototypes disperse more uniformly between the phases.

Polymer Blends Compound Data

Polymer system	50/50 NR/BR				50/50 NR/SBR			
Filler	Carbon Black	Control 1	Control 2	LD0380-2	Carbon Black	Control 1	Control 2	LD0380-2
ML(1+4)	84	88	71	98	91	83	72	73
Dispersion (%)	89	82	93	97	71	77	87	78
Tensile, MPa	22.0	21.3	16.1	23.6	25.5	26.5	20.9	24.8
Elongation, (%)	298	525	594	631	309	588	515	565
Hardness @ 23 °C	76	70	61	69	74	71	65	70
Rebound @ 23 °C	55	58	53	57	44	51	53	53
G' @ 60 °C, MPa	5.0	4.3	1.6	4.8	4.0	3.3	1.8	3.2
tan (δ) @ 60 °C	0.146	0.098	0.134	0.106	0.142	0.121	0.117	0.093
G' @ 0.5 %, MPa	8.8	5.8	2.5	6.3	6.9	4.7	2.7	4.3
Δ G', 0.5 % - 16 %	5.5	3.1	0.9	3.4	3.9	2.2	0.8	1.8
tan (δ) @ 1.0 %, 30°C	0.131	0.081	0.159	0.089	0.134	0.109	0.132	0.083
DIN Abrasion Index	100	127	131	128	74	71	92	73
Streblor Tear (N/mm)	17.3	34.3	43.5	42.6	20.5	33.8	25.0	38.3

- Silica prototypes showed improved lab compound indicators vs. control samples
- Dispersion improved $\approx 10\%$
- Further compound adjustments needed in Phase 2

Responses to Reviewer Comments

- **Tires for different applications are likely to require different properties. Should have identified primary target.**
 - Class 8 and Class 7 truck tires. Also target for the EPA SmartWay program. Few tire sizes will allow rapid implementation.
- **Establish an intermediate metric to track progress on RR reduction**
 - Milestone 3.1.: One tread compound formula with a >40% reduction in $\tan \delta$ at 60°C with no more than a 20% reduction in hardness, tear strength, or laboratory abrasion.
 - Phase 2 Go/No go decision: one TBR tread compound formula with a >20% decrease in RR measured on tire, and +/- 10% hardness, tear strength, and treadwear.
- **It is not clear what BATO brings. It would have been advisable to bring a trucking company to advise on the different type of use conditions.**
 - Bridgestone largest tire manufacturer in the world. BATO selected tread compounds and tire casings. Advised on selected silica prototypes to move forward and currently developing mixing conditions. Will build and test tires.
- **It is unclear how this work will demonstrate 4-6% savings in fuel efficiency.**
 - BATO is going to build tires with experimental and control treads and measure on-road fuel efficiency. We will also provide tires to the DOE for independent testing.

Collaboration and Coordination with Other Institutions

- **Bridgestone Americas Tire Operations (subrecipient)**
 - Working as advisor for the duration of the program
 - Provides truck tire tread compound formulations
 - Currently performing mixing optimization of silica in NR-based compounds
 - Will perform tire builds and tire testing at end of BP2 and BP 3.
- **Augustine Scientific**
 - State of the art surface energy measurement laboratory
 - Conducted measurements of rubber surface tension using polar and non-polar liquids at room and molten-state temperatures
 - Conducted measurements of silica controls and treated silicas using polar and non-polar liquids

Remaining Challenges and Barriers

- **Depending on the homogeneity of rubber blends, a single silica solution may not work**
 - Look at blends of treated silicas or mixing techniques
- **Current TBR formulations are optimized for carbon black**
 - BATO is optimizing TBR formulations for silica prototypes
- **Mixing conditions optimization**
 - BATO developing optimum conditions for prototype silica/NR mixing
- **Compounding Observations**
 - Selected samples demonstrated improved dispersion compared to carbon black and untreated silica in most of the single polymer or polymer blend-based compounds. We believe that tire rolling resistance will improve due to reduced hysteresis observed in the sample compounds
 - Rolling resistance and laboratory data ($\tan \delta$) targets represent our best estimation of the values necessary to achieve the project goals for improved fuel efficiency.

Proposed Future Work

- **Within Project**

- Silica Materials

- ✦ Develop understanding of how level of surface treatment should scale with surface area
 - ✦ Goal: Maximize tread wear, rolling resistance and tear strength
 - ✦ Optimize key metrics: processing, cure, dispersion, stress-strain, dynamic properties

- Rubber Compounds

- ✦ Develop optimized TBR compound, and mixing parameters, containing prototype silicas.
 - ✦ Perform tire build and testing by the end of March 2017

- **Beyond Project**

- Evaluation of advanced fillers in non-tread portion of tires
 - Market development for truck retread market segment

Summary

- **Objective**

- Develop a novel surface-modified silica technology and demonstrate 4-6% improved fuel efficiency of TBR tires built from the technology

- **Accomplishments**

- Determined surface energy of treated silicas and rubbers
- Directly observed the distribution of different fillers in binary polymer blends.
- Lab Compound data shows promising performance for prototype silicas

- **BP2 Goals: Improved Tire Compound Performance**

- Identify at least one TBR tread compound formula with
 - ✦ >20% decrease in rolling resistance as measured on-tire
 - ✦ +/- 10% hardness, tear strength, and tread wear compared to a carbon black-filled natural rubber-based control when measured on-tire



Back-up

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Approach

- **Budget Period 1: CONTROLLING DISPERSION:**

- ✓ Understand how different silica surface chemistries and surface areas are linked to dispersion performance in different rubber phases including both natural and synthetic rubbers
- ✓ Selection of silica chemistries and surface areas that deliver the most consistent dispersions in various rubbers

- **Budget Period 2: DEVELOP NEW TREAD COMPOUND:**

- Use the design principles identified in BP 1 to reduce the rolling resistance of a TBR compound by at least 60% compared to carbon black with no decrease in hardness and equal or better tear strength and tread wear
- Select no more than two combinations of silica and rubber formulations to make an experimental tire build and conduct on-tire testing to identify any performance gaps

- **Budget Period 3: OPTIMIZING FORMULA FOR ON-TIRE PERFORMANCE:**

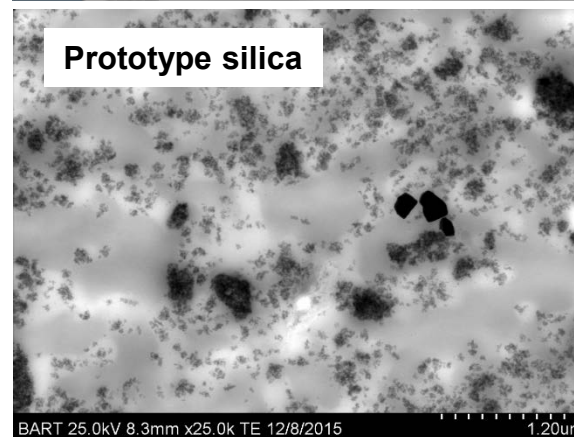
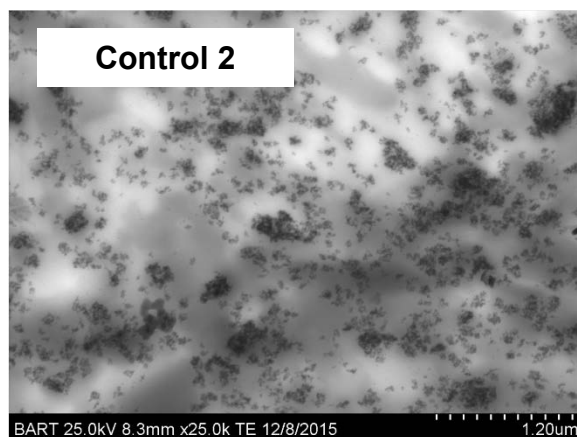
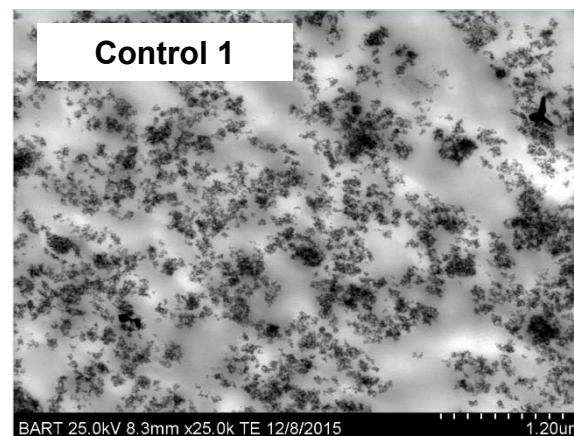
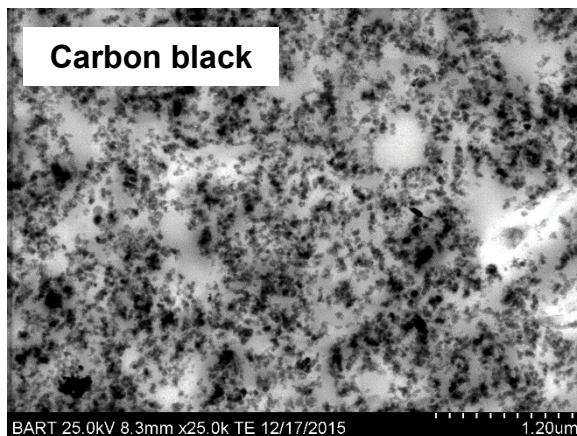
- Optimize the TBR compound performance and select the final rubber compound formulations for the tire builds that will be delivered to DOE for independent testing

Budget Period 2

- **Task 3 - Developing an Improved TBR Compound at Bench Scale**
 - Develop TBR tread compounds containing up to three of the silicas and polymer blends to deliver a >40% reduction in rolling resistance as measured by $\tan \delta$ at 60°C compared to an all carbon black-filled natural rubber-based compound
 - Hardness, tear strength, and tread wear will be evaluated at bench scale
- **Task 4 - Experimental Tire Build**
 - Generate on-tire data to identify performance gaps
 - Subtask 4.1: Produce Novel Silica at Pilot Scale
 - ✦ Produce approximately 500 lbs. each of up to three silica compositions
 - Subtask 4.2: Produce Novel Tread Compounds and Tires
 - ✦ The silicas will be compounded in up to two TBR tread formulations
 - ✦ Up to two novel formulations and one control formulation will be used to tread approximately 30 test tires
- **Task 5: On-Tire Testing**
 - Generate on-tire data to identify any performance gaps

Polymer Blends - SEM Microscopy Results

Dispersion in NR/BR Polymer Blends



BR: dark phase; NR: white phase

- Microscopy data agrees with NMR.

Dispersion Indicators – Selected Silica Prototypes

- Silica prototypes with low surface energy selected
- NR, 60phr silica, 4.8phr Si266 in non-treated silica.

Compound	Filler	Carbon Black	Control 1	Control 2	LD0380-1	LD0380-2	LD0381-1	LD0382-1
100phr NR	Dispersion (%)	87.9	98.5	99.4	99.3	96.9	98.7	99.4
	$\Delta G'$, 0.5 % - 16 %	7.9	4.3	1.8	1.8	4.8	2.7	1.2
100phr BR	Dispersion (%)	98.2	99.0	98.5	95.0	94.5	96.5	99.7
	$\Delta G'$, 0.5 % - 16 %	6.2	3.8	2.2	3.6	4.9	3.5	1.0
100phr SSBR	Dispersion (%)	96.9	96.6	99.2	97.4	92.1	94.1	99.3
	$\Delta G'$, 0.5 % - 16 %	3.9	3.3	1.2	1.5	4.6	2.4	0.5

- Silica prototypes showed improved dispersion by Dispergrader and Payne effect
- Silica prototypes with lowest surface energy selected to move forward.