Production of High Value Products from Coal
U.S. Coal to Conductive Inks

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### Fossil Energy Objective

- Increase use of U.S. coal utilization through the development of technologies and value-added products that use U.S. Coal Supplies as a primary feedstock

### Minus 100, LLC Value Proposition

- Secure/Abundant Feedstocks
- Increased Wettability
- Lower Curing Temperatures
- Lower Cost
- Lower Particle Density
- Low Resistance/Resistivity
- Minus 100's T-Series
- Higher Solids Loadings
Conductive Pigment Markets

• Conductive Inks
  • Current Market (~ $ 3.0 B/yr) Growing at 4-8% CAGR
  • Conductivity Enhancement Methods Under Evaluation
  • Lower Resistivity Leads to Electronic Printing Market Expansion

• Conductive Paints & Coatings
  • Significantly Larger Market (~ $ 17.5 B/yr) Growing at 6.8% CAGR
  • Application Methods are Simpler

• Synthetic Graphite Manufacturing
  • Significantly Larger Market (~ $ 17.4 B/yr) Growing at 5.2% CAGR
  • Precursor to Various Carbon Allotropes
    • Graphene, Carbon Nanotubes
  • Strategic Material with Limited U.S. Manufacturing Capacity

• Underfloor Heating Market
  • (~$6 B/yr) Growing at 4.5% CAGR
Technical & Commercialization Accomplishments

Technical Accomplishments/Activities

Conductive Ink Sheet Resistivity Performance Commercialization Activities

• Accomplished Phase II Objective of ≤ 100 ohm/sq/mil
• Major Ink Manufacturers are Evaluating Coal-Based Pigment & Ink Suspensions for Conductive Ink Applications
• Working Toward Qualification of our Pigments/Suspensions for Specific Commercial Applications
• Testing of Lab-scale Proto-type Heating Element Assemblies
• Working with Screen Printers to Scale-up Heating Element Assembly Designs
• Developing Lower Resistivity Pigments/Suspensions to Expand the Application Scope of our Coal-Based Pigments/Suspensions
# Carbon-Based Conductive Ink Applications

## Sheet Resistivity

<table>
<thead>
<tr>
<th>Sheet Resistivity (ohm/sq/mil)</th>
<th>Printing Method</th>
<th>Potential Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,000,000 ± 15%</td>
<td>Screen-printing, thermoset, rigid carbon ink, blending for specific resistance targets</td>
<td>Printed resistors, heaters, potentiometers, friction</td>
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<tr>
<td>&lt; 40</td>
<td>Flexographic, Gravure, Screen, Digital</td>
<td>Printed resistors, heaters, potentiometers, friction</td>
</tr>
<tr>
<td>1</td>
<td>Flexographic, Gravure, Screen, Digital</td>
<td>RFID Applications</td>
</tr>
</tbody>
</table>
Position of Minus 100 in the Printing Value Chain

Minus 100 Position within the Printing Value Chain & Important Pigment/Ink Parameters

<table>
<thead>
<tr>
<th>Minus 100 Position within the Printing Value Chain</th>
<th>Important Performance Parameters of Conductive Carbon Pigments Inks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw Material Sourcing</td>
<td><strong>Pigment Properties</strong></td>
</tr>
<tr>
<td>Intermediate Feedstock Suppliers</td>
<td>• Sheet/Volume Resistivity (ohm/sq), (ohm/sq/mil)</td>
</tr>
<tr>
<td>Ink Formulators</td>
<td>• Powder Resistivity (ohm-cm)</td>
</tr>
<tr>
<td>Printing &amp; Packaging Companies</td>
<td>• Particle Size (μm)</td>
</tr>
<tr>
<td></td>
<td>• Surface Area (m²/g)</td>
</tr>
<tr>
<td></td>
<td>• Density (g/cc)</td>
</tr>
</tbody>
</table>

**Ink Properties**

- Viscosity (Pa·s)
- Surface Tension (dyne/cm)
- Substrate Surface Energy & Surface Adhesion
- Curing/Drying Rate & Temperature
- Abrasion Resistance
- Flexibility
- Color/Transparency
- Toxicity

Typically Mined Materials, Domestic Raw Material Supply Eliminates Reliance on Uncertain Global Conditions

Pigments/Suspensions Base Vehicles & Additives (e.g., Binders, Coatings, Preservatives, Dispersants, Curing Agents)

Formulates Inks in Accordance with Printing Method Utilized and Substrate to be Utilized Paper & Cardboard Plastic & Metals Lithographic Printing Flexographic Printing Gravure Printing Screen Printing Inkjet Ink Printing
Technical Approach

- Coal-Based Feedstock Selection
- Communion
- Thermal Treatment
- Electrochemical Processing
- Mechanical Processing
- Ink Formulation
- Product Evaluation and Qualification
Proprietary Thermal Treatment

- Elevated temperatures enhance the electrical conductivity of coal-based feedstocks.
- Minus 100, LLC has developed a novel high temperature process for converting coal-based feedstocks into electrically conductive materials.
- This process is suitable for the production of synthetic graphite, a strategic material, from coal-based feedstocks.
- Patent application restrictions preclude disclosure of details.
Enhanced Conductivity of Bituminous Feedstocks via Thermal Treatment

![Image of equipment and graph showing powder resistivity vs. applied pressure for different samples: PC4876-N, PC4876, and HTPC4876.]
Technology/Product Development Approach

Ink Resistivity Measurements

- Draw Down with a Mayer Rod
- Measurement of Square Resistance
- Measurement of Ink Film Thickness
Lab-Scale Resistive Heating Element Testing Underway

Coal-Based Heating Element Assembly Test Arrangement

Lab Scale Under Floor Heating Element Assembly

Thermographic Image of Lab-Scale Heating Assembly

Thermoprofile of Lab-Scale Heating Element Assembly

Temperature (°C) vs Applied DC Voltage for a Selected Coal-based Pigment/Ink
# Carbon Additive Enhancement of Coal-Based Pigments

## Description of Electro-chemical Reactor Assembly

<table>
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<tr>
<th>Electrochemical Reactor for Processing Carbon Powders</th>
<th>Intercalation-Exfoliation Mechanism</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rotating Anode</td>
<td>Graphene</td>
</tr>
<tr>
<td>DC Power Source</td>
<td>Sulfate Intercalation</td>
</tr>
<tr>
<td>Cathode</td>
<td>0.33nm</td>
</tr>
<tr>
<td>Collection Reservoir</td>
<td>Electrical Exfoliation</td>
</tr>
<tr>
<td>4 liter cell</td>
<td>0.66nm</td>
</tr>
</tbody>
</table>

**Graphite**

**Exfoliated Effluent**

**Graphene**

**Sulfate Intercalation**

**Electrical Exfoliation**

**DC**

**Rotating Anode**

**Cathode**

**Collection Reservoir**

**4 liter cell**

**DC Power Source**

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**Electrical Exfoliation**

**DC**
Additive Enhancement for Coal-Based Pigments

Experimental Design (DOE) for the Production of Graphite/Graphene Platelets

Factor (X) | Levels | Experimental Trials | Results (Y)
--- | --- | --- | ---
Graphite Platelets | 5wt% 10wt% | All possible level combinations yield 12 trials. | Sheet Resistivity
Na₂SO₄ | 0.5M 1.0M | Design results will be randomized and analyzed using Minitab Software | Powder Resistivity
Intercalation Time @ 2.00V | 10min 20min 40min | | Particle Size
 | | | Expansion Fraction
Additive Enhancement for Coal-Based Pigments

Preliminary Analysis for Electrochemical Reactor DOE

- Residual Carbon Fraction Analysis
- Main Effects Carbon Fraction Analysis
  - Lower carbon% = lower sheet resistivity
  - Higher intercalation time = lower sheet resistivity
  - Higher salt concentration = lower sheet resistivity
- Effluent Carbon Fraction Analysis in process with target date 10/31/2020.
Additive Enhancement for Coal-Based Pigments

**Metallization of Coal-Based Pigments via Electroless Nickel Plating**

<table>
<thead>
<tr>
<th>Property / Level of alloy</th>
<th>High $^a$</th>
<th>Mid $^a$</th>
<th>Mid-Low $^a$</th>
<th>Low $^a$</th>
</tr>
</thead>
<tbody>
<tr>
<td>% Phosphorous</td>
<td>10 - 13</td>
<td>7 - 9</td>
<td>4 - 6</td>
<td>1 - 3</td>
</tr>
<tr>
<td>Electrical resistivity $^b$</td>
<td>75 - 110</td>
<td>40 - 70</td>
<td>15 - 45</td>
<td>10 - 30</td>
</tr>
</tbody>
</table>

$^{2}Ni^{2+} + 8H_{2}PO_{4}^{-} + 2H_{2}O \rightarrow 2Ni_{0} (s) + 6H_{2}PO_{4}^{-} + 2H^{+} + 2P (s) + 3H_{2} (g)$

**Stage I**
- 5µm coal based conductive particles were successfully coated with nickel alloy.
- Lowers overall weight and cost compared to silver.

**Stage II**
- NiP alloy coating with targeted resistivity 15-45 µohm-cm range
SWOT Analysis

Strengths
• Phase I Goal: < 1000 ohm/sq/mil - Achieved
• Phase II Goal: < 100 ohm/sq/mil - Achieved
• Internal Goal: < 10 ohm/sq/mil – Achieved 8 ohm/sq/mil
• Next Target: ≤ 1 ohm/sq/mil – In Progress
• Lab-Scale heating element prototype developed
• Development of novel high temperature process for conductive/graphitic enhancement
• Use of nontoxic electrolytes in ECR

Challenges
• Achieve ≤ 1 ohm/sq/mil with metallized pigment.
• HT Furnace construction materials
• Flue Gas Emissions
• Material handling of fine particles

Opportunities
• Seeking underfloor heating commercialization partner
• New electronic circuit printing
• Collaborating with major ink manufacturers
• Conductive Paints and Coatings
• Synthetic Graphite Production
• Semiconductor Chemical Mechanical Planarization (CMP) Spillover

Threats
• Covid-19 Fallout (supply and co-development)
• Demand destruction
Future Plans

1. Underfloor Heating Element Applications
   • Pursuing co-development commercial screen-printing partners to produce a prototype.
2. Electro-Chemical Research
   • Complete Design of Experiment analysis to target optimum ECR conditions.
   • Evaluate intercalation/exfoliation potential of selected coal-based pigments with optimum ECR conditions
3. Hybrid Pigment Conductive Research
   • Complete carbon-based additive research
     • Conductive Carbon Black
     • Graphite/Graphene Platelets
     • Carbon Nanotubes
     • Metallization
4. Continue collaboration with major ink formulators to evaluate Minus 100, LLC conductive pigments.
5. Initiate lab-scale testing and evaluation of proprietary heating technology at elevated temperatures
6. Continue commercialization efforts with Tech-Opps.