Lunar Construction Material Production Using Regolith Simulant in a Geothermite© Reaction

Eric J. Faierson and Kathryn V. Logan
Virginia Polytechnic Institute and State University-National Institute of Aerospace
Materials Science and Engineering

2009 Annual Meeting of the Lunar Exploration Analysis Group
Background
Geothermite© Reactions

• Minerals + reducing agent
  – Thermite/SHS reaction behavior
• SHS → Self-propagating High-temperature Synthesis
  • Requires initial energy input
    • Sufficient to initiate reaction
  • Once reaction starts, no further external energy needed, continues until reactant fuel is consumed
• Variables
  • Particle Size
  • Compaction
  • Stoichiometry
  • Atmosphere and Pressure
Regolith Geothermite© Reaction

- Lunar regolith simulant + Al powder

- Regolith Simulant
  - JSC-1AF and JSC-1A
  - Commissioned by NASA
  - Volcanic ash deposit in AZ
  - Composed of minerals and glass
    - Primary Components
      - Plagioclase Solid Solution Series
      - Basaltic Glass

(NASA MSFC, 2006)
Constituents of Lunar Regolith and Simulant JSC-1AF in weight %

<table>
<thead>
<tr>
<th>Constituents</th>
<th>Maria</th>
<th>Highlands</th>
<th>Simulant</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Apollo 14</td>
<td>Luna 16</td>
<td>Luna 20</td>
</tr>
<tr>
<td>SiO$_2$</td>
<td>47.93</td>
<td>41.70</td>
<td>45.40</td>
</tr>
<tr>
<td>TiO$_2$</td>
<td>1.74</td>
<td>3.38</td>
<td>.47</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>17.6</td>
<td>15.33</td>
<td>23.44</td>
</tr>
<tr>
<td>FeO</td>
<td>10.37</td>
<td>16.64</td>
<td>7.37</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>MgO</td>
<td>9.24</td>
<td>8.78</td>
<td>9.19</td>
</tr>
<tr>
<td>CaO</td>
<td>11.19</td>
<td>12.50</td>
<td>13.38</td>
</tr>
</tbody>
</table>

(Sen, Ray, and Reddy, 2002); (NASA MSFC, 2006)
Experimental
Reaction Parameters

• Simulant Particle Size
  – JSC-1AF: mean ~25 µm (NASA MSFC, 2006)
  – JSC-1A: mean ~185 µm (NASA MSFC, 2007)

• Environment
  – Ambient
  – Vacuum (0.600 Torr)

• Reactant Stoichiometries (wt.%)
  – 80.56% Simulant / 19.44% Al
  – 75.55% Simulant / 24.45% Al
  – 71.15% Simulant / 28.85% Al
  – 66.67% Simulant / 33.33% Al
Sample Preparation

- 80g Mixtures of Simulant and Aluminum
  - Aluminum (-325 mesh) (<44 µm)
  - Manually Mixed

- Aluminum Foil Cylinder Crucible (~1” dia.)
  - Mixture poured in fifths
  - Tamping after each fifth
  - Wire used to disperse layering

- 12” NiCr wire
  - Portion immersed fully in sample
  - Wire ends connected to terminals
  - Sample tamped to decrease particle disturbance
Ambient Reaction Initiation

- Reaction chamber lined with refractory brick
- Variac power supply
  - Standard Rate of Power Application
    - Increase current to ~22A over 4.5 minutes
    - Held at ~22A until reaction initiation
    - Reaction initiation causes break in NiCr wire
    - No further external energy applied
Vacuum Reaction Initiation

- Vacuum chamber with refractory bricks
  - Vacuum traps
  - 30 ampere electrical feedthrough
- Double-braided NiCr wire
  - Changed resistance and current applied for equivalent Variac settings
- Variac power supply
  - Standard Rate of Current Application
    - Increase current to ~22A over 0.5 minutes
    - Held at ~22A until reaction initiation
      - Continuous vacuum pumpdown
Materials Characterization

• X-ray diffraction
• Scanning Electron Microscopy
  – Fracture Surfaces
• Compressive Strength
  – Dry cut using ceramic tile saw with diamond blade
Results and Discussion
Ambient Reaction Process

Heat Applied

Reaction Initiation (RI)

~ 2 min after RI

~ 2 min 30 sec after RI

~ 3 min 10 sec after RI

~5 min 40 sec after RI
Vacuum Reaction Process

Heat Applied

~ 40 sec after RI

Reaction Initiation (RI)

~ 3 min after RI

~ 4 min after RI

~ 20 sec after RI

~6 min after RI
X-Ray Diffraction

• Standard Atmosphere Chemical Species
  – Silicon
  – Corundum (Al$_2$O$_3$)
  – Spinel (MgAl$_2$O$_4$)
  – Grossite (CaAl$_4$O$_7$)
  – Aluminum Nitrides
  – Iron Silicides

• Vacuum Differences
  – No nitrides
  – Unreacted minerals from simulant, unreacted Al
SEM/EDS: Ambient Nanostructures

<table>
<thead>
<tr>
<th>Element</th>
<th>Wt%</th>
<th>Atm%</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>49.80</td>
<td>63.05</td>
</tr>
<tr>
<td>Na</td>
<td>0.99</td>
<td>0.87</td>
</tr>
<tr>
<td>Mg</td>
<td>4.51</td>
<td>3.75</td>
</tr>
<tr>
<td>Al</td>
<td>35.98</td>
<td>27.01</td>
</tr>
<tr>
<td>Si</td>
<td>5.19</td>
<td>3.74</td>
</tr>
<tr>
<td>Ca</td>
<td>2.03</td>
<td>1.03</td>
</tr>
<tr>
<td>Fe</td>
<td>1.51</td>
<td>0.55</td>
</tr>
</tbody>
</table>
SEM/EDS: Ambient Nanostructures

<table>
<thead>
<tr>
<th>Element</th>
<th>Spectrum 1 Wt%</th>
<th>Spectrum 1 Atm%</th>
<th>Spectrum 2 Wt%</th>
<th>Spectrum 2 Atm%</th>
</tr>
</thead>
<tbody>
<tr>
<td>N</td>
<td>19.34</td>
<td>30.31</td>
<td>22.12</td>
<td>33.47</td>
</tr>
<tr>
<td>O</td>
<td>9.55</td>
<td>13.10</td>
<td>11.57</td>
<td>15.32</td>
</tr>
<tr>
<td>Mg</td>
<td>-</td>
<td>-</td>
<td>1.28</td>
<td>1.12</td>
</tr>
<tr>
<td>Al</td>
<td>64.35</td>
<td>52.34</td>
<td>55.11</td>
<td>43.29</td>
</tr>
<tr>
<td>Si</td>
<td>3.77</td>
<td>2.94</td>
<td>8.09</td>
<td>6.11</td>
</tr>
<tr>
<td>Ca</td>
<td>0.81</td>
<td>0.44</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Fe</td>
<td>2.18</td>
<td>0.86</td>
<td>1.82</td>
<td>0.69</td>
</tr>
</tbody>
</table>
Vacuum Microstructures
### EDS: Vacuum Microstructures

<table>
<thead>
<tr>
<th>Element</th>
<th>Spectrum 1</th>
<th>Spectrum 2</th>
<th>Spectrum 3</th>
<th>Spectrum 4</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Wt%</td>
<td>Atm%</td>
<td>Wt%</td>
<td>Atm%</td>
</tr>
<tr>
<td>O</td>
<td>8.45</td>
<td>16.99</td>
<td>31.87</td>
<td>43.97</td>
</tr>
<tr>
<td>Mg</td>
<td>-</td>
<td>-</td>
<td>15.42</td>
<td>14.00</td>
</tr>
<tr>
<td>Al</td>
<td>2.89</td>
<td>3.43</td>
<td>45.03</td>
<td>36.84</td>
</tr>
<tr>
<td>Si</td>
<td>49.80</td>
<td>57.09</td>
<td>4.11</td>
<td>3.23</td>
</tr>
<tr>
<td>Ca</td>
<td>0.40</td>
<td>0.32</td>
<td>3.56</td>
<td>1.96</td>
</tr>
<tr>
<td>Ti</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Mn</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Fe</td>
<td>38.46</td>
<td>22.17</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
## EDS: Vacuum Microstructures

![Image of vacuum microstructures](image_url)

### EDS Table

<table>
<thead>
<tr>
<th>Element</th>
<th>Spectrum 1</th>
<th>Spectrum 2</th>
<th>Spectrum 3</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Wt%</td>
<td>Atm%</td>
<td>Wt%</td>
</tr>
<tr>
<td>O</td>
<td>43.19</td>
<td>57.88</td>
<td>50.52</td>
</tr>
<tr>
<td>Mg</td>
<td>1.84</td>
<td>1.62</td>
<td>1.47</td>
</tr>
<tr>
<td>Al</td>
<td>39.60</td>
<td>31.46</td>
<td>33.54</td>
</tr>
<tr>
<td>Si</td>
<td>4.81</td>
<td>3.66</td>
<td>5.51</td>
</tr>
<tr>
<td>Ca</td>
<td>8.65</td>
<td>4.63</td>
<td>7.22</td>
</tr>
<tr>
<td>Ti</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Fe</td>
<td>1.91</td>
<td>0.73</td>
<td>1.75</td>
</tr>
</tbody>
</table>
Mean Compressive Strength of JSC-1A and JSC-1AF Ambient Reaction Products

Blue: Coarse (JSC-1A)
Orange: Fine (JSC-1AF)
Potential Applications
Potential Applications

• Auxiliary Lunar Physical Asset/Habitat Construction Material
  – Structural
  – Radiation Shielding
  – Micro-meteoroid protection

• Lunar dust mitigation
  – Lunar landing sites
  – Roads
  – Blast Berms

• Mineral Processing

• Thermal energy
Construction Methods

A potential habitat architecture that is expandable

Computer generated images courtesy of Brian Stewart, NASA LaRC
Near-net shape
Ambient Near-net Shape Reaction Video (2x)

Video Credit: NIA Media Team
Conclusions
Conclusions

- Geothermite reaction between JSC-1A/AF and Al occurred
  - Range of reactant stoichiometries
  - Ambient: Produced coherent near net shaped ceramic-composite material
  - Vacuum: Semi-coherent material
    - Better synthesis methods need to be investigated
- Si, Corundum (Al₂O₃), grossite (CaAl₄O₇), and spinel (MgAl₂O₄) were common species in atmospheric and vacuum reactions
Conclusions

• Whiskers of aluminum nitrides and aluminum oxides formed in ambient conditions

• Highest compressive strengths produced by lowest Al stoichiometries in JSC-1A simulant (~18 MPa, 2600 psi)
Conclusions-XRD Vacuum Synthesis

• Extent of reaction
  – As Al increased in reactants, the quantity of unreacted simulant minerals decreased in the product
  – Larger quantities of unreacted minerals tended to exist in the JSC-1A product

• No AlN formation
Future Work
Future Work

• Reaction Initiation
  – Microwaves
  – Concentrated Solar Flux
• Other Reducing Agents
  – Al-Li, Carbon
• Radiation Transmission Characteristics
• Pressure/Gas whisker relationships
• Temperature profiles for various reaction parameters
• Press reactants
  – Maximize particle contact and decrease porosity
Future Work-Mars Regolith

Mars Regolith Composition (Wt. %)

<table>
<thead>
<tr>
<th></th>
<th>VL-1</th>
<th>VL-2</th>
<th>Pathfinder</th>
<th>JSC-1AF</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO$_2$</td>
<td>43</td>
<td>43</td>
<td>44.0</td>
<td>47.1</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>18.5</td>
<td>17.8</td>
<td>16.5</td>
<td>3.41 (7.57)</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>7.3</td>
<td>7</td>
<td>7.5</td>
<td>17.1</td>
</tr>
<tr>
<td>MgO</td>
<td>6</td>
<td>6</td>
<td>7.0</td>
<td>6.9</td>
</tr>
<tr>
<td>CaO</td>
<td>5.9</td>
<td>5.7</td>
<td>5.6</td>
<td>10.3</td>
</tr>
<tr>
<td>SO$_3$</td>
<td>6.6</td>
<td>8.1</td>
<td>4.9</td>
<td>-</td>
</tr>
<tr>
<td>TiO$_2$</td>
<td>0.66</td>
<td>0.56</td>
<td>1.1</td>
<td>1.87</td>
</tr>
</tbody>
</table>

Allen, et.al, MARTIAN REGOLITH SIMULANT JSC MARS-1.
Acknowledgements

• NIA contract VT-03-01 for funding this work
• NASA Langley Research Center for the use of laboratory facilities
• Jim Baughman, at LaRC, and Steve McCartney, at ICTAS for assistance and training with SEM
• David Hartman, for assistance with XRD at LaRC
• Mac McCord at Virginia Tech for assistance with compressive strength testing
• Dr. Wallace Vaughn and Craig Leggette in the Carbon-Carbon Lab at LaRC for assistance with compressive strength testing
• Center for Multi-functional Aerospace Materials
Questions?