Characterization Strategies and Requirements for Lunar Regolith Simulant Materials

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Outline

• Development of LRSM and characterization
  Bulk chemical and microanalysis methods
• Lunar regolith simulants JSC-1 and MLS-1
  Bulk chemistry and electron-probe microanalysis data
• Root simulant development strategies
  JSC-1 example
  Japanese simulants FJS-1, Type 1-3
  Synthetic agglutinate microspheres, status report
• Quality control and standard development
• Oxygen fugacity control and Mössbauer characterization
• Source localities for anorthosite simulants
Development of LRSM and Characterization

LRSM development sequence
Bulk chemical analysis methods
Microchemical / structural analysis methods
Development and Distribution of LRSM: Role of Characterization

• Identification of source materials, localities, mines, synthetic materials relative to requirements
• Initial batching for test screen evaluation: processing and required physical, chemical, mineralogical characterization
• Evaluation of initial screen relative to requirements list
• Batch production quantity, processing, synthesis, mixing
• Characterization and quality control of master simulant batch
• Subdivision for distribution, quality control of sub-batches
• Curation, storage, shelf monitoring
• Guidelines for simulant use
• Requests for simulant
• Oversight by monitoring committee
Characterization Requirements for Lunar Regolith Simulant Materials

• Bulk and microchemical analysis required to support physical and chemical characterization of LRSM
  • **Bulk analysis**: large/many particles, representative sample from parent XRF, ICPMS, INAA, XRD, Mössbauer, many others
  • **Microanalysis**: few/individual, analysis of dust, grain, rock fragments EPMA, SEM, TEM, SIMS, optical microscopy, others
• Calibration and quality control of LRSM root and composite simulant reference “standards” for development and distribution
• Diverse set of lunar materials requires relatively diverse set of LRSM—there is no single simulant that represents the spectrum of lunar materials
• Anticipate need for real-time autonomous analysis for material selection, process monitoring, error recovery, etc. on planetary surfaces
• Characterization is part of overall strategy on Earth, moon, Mars
Characterization Methods: Electron-probe microanalysis / SIMS

• EPMA Electron-probe microanalysis / SEM Scanning electron microscopy / TEM Transmission electron microscopy
  Nearly all lunar samples analyzed using EPMA / SEM
  Polished, conductive sample, 1 μm spatial resolution, vacuum technique
  Analytical sensitivity:
  Wavelength-dispersive spectrometer 0.x mg/g to x μg/g sensitivity, serial
  Energy-dispersive spectrometer 0.x mg/g best case, parallel
  New Si-drift detector with digital electronics ~ WDS performance
  Micro and bulk analysis capabilities with high-speed sample mapping

• SIMS Secondary-ion mass spectrometry
  Recent use for spatial trace element analysis beyond EPMA sensitivity
  Polished, conductive sample, ~1-10 μm spatial resolution, vacuum technique
  Analytical sensitivity μg/g to ng/g but calibration requires bracketing stds
Lunar Regolith Simulants JSC-1 and MLS-1

Bulk chemistry and target Apollo soils
Electron-probe microanalysis characterization
Lunar Mare Basaltic Simulants: JSC-1, MLS-1 and Apollo Soils

Simulant Materials:

<table>
<thead>
<tr>
<th>Oxide</th>
<th>JSC-1</th>
<th>Apollo 14 Avg Soil</th>
<th>MLS-1</th>
<th>Apollo 11 Soil 10002</th>
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<td>SiO$_2$</td>
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<td>48.1</td>
<td>43.9</td>
<td>42.2</td>
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<td>1.7</td>
<td>6.30</td>
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<td>17.4</td>
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<td>13.6</td>
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<td>0.30</td>
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<tr>
<td>Fe$_2$O$_3$</td>
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<td></td>
<td>2.60</td>
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<tr>
<td>FeO</td>
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<td>10.4</td>
<td>13.40</td>
<td>15.3</td>
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<tr>
<td>MnO</td>
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<td>0.14</td>
<td>0.20</td>
<td>0.20</td>
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<tr>
<td>MgO</td>
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<td>9.4</td>
<td>6.70</td>
<td>7.8</td>
</tr>
<tr>
<td>CaO</td>
<td>10.42</td>
<td>10.7</td>
<td>10.10</td>
<td>11.9</td>
</tr>
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<td>Na$_2$O</td>
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<td>0.70</td>
<td>2.10</td>
<td>0.47</td>
</tr>
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<td>K$_2$O</td>
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<td>0.55</td>
<td>0.20</td>
<td>0.16</td>
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<tr>
<td>P$_2$O$_5$</td>
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<td>0.51</td>
<td>0.13</td>
<td>0.05</td>
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<tr>
<td>LOI</td>
<td>0.71</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Total</td>
<td>99.65</td>
<td>99.8</td>
<td>99.20</td>
<td>99.9</td>
</tr>
</tbody>
</table>

Apollo data from McKay et al., chapter 7, Lunar Sourcebook, Table 7.15, p. 346
Comparison of JSC-1 and MLS-1: EPMA Backscattered-electron Stage Maps

L: JSC-1 BSE digital image  R: MLS-1 BSE digital image
Polished mount, large area map. BSE contrast is function of average Z.
Image segmentation yields phase area fraction, convert to weight fraction.
Resolution ~1 μm, acquisition time beam ~ minutes, stage ~ hours.
JSC-1: BSE images
Lithic fragments and monomineralic grains

JSC-1 BSE digital image, beam maps (L) 100x mag, (R) 300x mag, enhanced contrast
Dark to bright: plagioclase, glass, olivine, Ca-pyroxene, ilmenite, magnetite
Expect coarse size plagioclase dominant, fine size oxide, olivine, cpx, glass
JSC-1 vs. MLS-1:
BSE images, 100x Magnification

JSC-1: Finer lithic and grain size. Expect lithics to persist to finer fraction vs. MLS-1.
MLS-1: Grain size issue apparent, ~ 5x larger max grain size than JSC-1. Ilmenite mode greater (high Ti), Ca-pyroxene with cpx lamellae, plagioclase secondary crystallization, absence of glass. Monomineralic grains at coarse size. Dark to bright: plagioclase, cpx, olivine, ilmenite, magnetite.
Lunar Simulant Mineral Chemistry  
Electron Microprobe, XRD

• **JSC-1:**
  Plagioclase An$_{64-71}$Ab$_{28-33}$Or$_{1-3}$
  Olivine Fo$_{82-86}$
  Ca-pyroxene Wo$_{45}$ En$_{34-38}$ Fs$_{17-21}$
  Ilmenite $\sim$ Fe$^{2+}_{0.85}$ Fe$^{3+}_{0.09}$ (MgMn)$_{0.103}$ (AlCr)$_{0.0025}$ Ti$_{0.95}$ O$_3$
  Other: Chromite, Clay, Cristobalite?, Magnetite?, Hematite?

• **MLS-1:**
  Plagioclase An$_{44-50}$ Ab$_{46-60}$ Or$_{3-5}$
  Olivine Fo$_{48-51}$
  Ca-pyroxene Wo$_{39-41}$ En$_{39-41}$ Fs$_{19-22}$
  Ilmenite $\sim$ Fe$^{2+}_{0.95}$ Fe$^{3+}_{0.0875}$ (MgMn)$_{0.075}$ (AlCr)$_{0.0025}$ Ti$_{0.95}$ O$_3$
  Magnetite-Ulvospinel $\sim$ Fe$^{2+}_{0.22}$ Fe$^{3+}_{2.05}$ (MgMn)$_{0.03}$ (AlCr)$_{0.12}$ Ti$_{0.25}$ O$_4$
EPMA: Micro vs. Macro Analysis
Quantitative EPMA point count grid

Left: Backscattered-electron digital image, contrast is function of average Z. Image segmentation yields phase area fraction, convert to weight fraction. Resolution ~1 μm, acquisition time ~ minutes.

Right: EPMA pc grid example. Grid spacing adjusted for sampling requirements. Resolution ~1 μm, acquisition time ~ hours-days.
MLS-1 Mineralogy BSE and Phase Map
Area Fraction of Mineral Composition

<table>
<thead>
<tr>
<th>Color</th>
<th>Phase</th>
<th>Area %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blue</td>
<td>Ca pyroxene</td>
<td>40</td>
</tr>
<tr>
<td>Red</td>
<td>Plagioclase</td>
<td>37</td>
</tr>
<tr>
<td>Yellow</td>
<td>Ilmenite</td>
<td>3.2</td>
</tr>
<tr>
<td>Green</td>
<td>Olivine</td>
<td>2.0</td>
</tr>
<tr>
<td>Cyan</td>
<td>Magnetite</td>
<td>0.9</td>
</tr>
<tr>
<td></td>
<td>Other</td>
<td>16.7</td>
</tr>
</tbody>
</table>

500 μm
Root Simulant Development Strategies

Root simulant materials and target Apollo soils
Least-squares bulk chemistry match
  JSC-1 example
  FJS-1 Japanese simulant examples
Synthetic Agglutinate-like microspheres, status
Calculation of Root Simulant Mix for Apollo 16 Target

- Example for Apollo 16 highland anorthosite target.
  \[ \text{SiO}_2 = (\% \text{ olivine})*41.23 + (\% \text{ plagioclase})*43.19 + (\% \text{ quartz})*100 = 46.29, \text{ etc.} \]
- Match using 16% olivine (Fo92), 78% plagioclase (An100), and 6% quartz, R=1.33
- CaO of target satisfied, but mix cannot match MgO and FeO simultaneously
- Pro’s: Evaluation of proposed roots, proportions, source materials
  Could use root basalt and mineral separates from that same root (JSC-1, MLS-1?)
- Con’s: Chemical match masks mineralogical requirements (but use mineral input)

<table>
<thead>
<tr>
<th>Oxide</th>
<th>Fo$_{92}$</th>
<th>An</th>
<th>Q</th>
<th>Root Mix</th>
<th>A 16*</th>
<th>Diff$^2$</th>
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</thead>
<tbody>
<tr>
<td>SiO$_2$</td>
<td>41.23</td>
<td>43.19</td>
<td>100</td>
<td>46.29</td>
<td>45.00</td>
<td>1.65</td>
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<tr>
<td>Al$_2$O$_3$</td>
<td>36.65</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FeO</td>
<td>7.89</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MgO</td>
<td>50.89</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>CaO</td>
<td>20.16</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>(100.78)</td>
<td></td>
</tr>
</tbody>
</table>

Sum of (L$_i$-C$_i$)$^2 = 24.75$, $R = \sqrt{24.75 / n=14 \text{ oxides}} = 1.33$

For mix of 16% olivine Fo$_{92}$, 78% Plagioclase An100, 6% quartz
Apollo 16 analysis includes other minor elements not listed here
Root Mixtures: JSC-1 and Component Minerals
Can JSC-1 be Used for Several Root Simulants?

• Root simulants using JSC-1 and JSC-1 mineral separates (ilmenite, olivine, Ca-pyroxene, plagioclase, glass). Mineral compositions from EPMA, ilmenite Fe²⁺/Fe³⁺ calculated by stoichiometry.
• For each proportion mix, determine least-squares match for all component oxides to compared to target Apollo oxide composition.

<table>
<thead>
<tr>
<th>Apollo Target</th>
<th>Apollo 11 High Ti</th>
<th>Apollo 14 Low Ti / KREEP</th>
<th>Apollo 16 Highland</th>
</tr>
</thead>
<tbody>
<tr>
<td>R min</td>
<td>1.68</td>
<td>1.41</td>
<td>1.43</td>
</tr>
<tr>
<td>JSC-1 %</td>
<td>86</td>
<td>61</td>
<td>50</td>
</tr>
<tr>
<td>Ilmenite %</td>
<td>14</td>
<td>12</td>
<td>15</td>
</tr>
<tr>
<td>Olivine %</td>
<td>2</td>
<td>5</td>
<td>4</td>
</tr>
<tr>
<td>Ca-pyroxene</td>
<td>15</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Plagioclase</td>
<td>10</td>
<td>8</td>
<td>10</td>
</tr>
<tr>
<td>Glass</td>
<td>22</td>
<td>13</td>
<td>2</td>
</tr>
</tbody>
</table>

Notes:
Apollo compositions from McKay et al., Chapter 7, Lunar Sourcebook, Table 7.15, p. 346.
Glass and Ca-pyroxene present separation challenge due to grain size in JSC-1 bulk material.
Minor phases and trace glass chemistry ignored.
Simulant Development in Japan
Mixtures from Root Materials

- Identify root components, iterate on component proportion (basalt + ilm + ol)
  For each proportion mix, determine least-squares match for all component oxides
  compared to target Apollo oxide composition.
- Mt. Fuji basalt, ilmenite from Florida, and olivine from Horoman and Hokkaido islands
  crushed and physically mixed.
- Best fit for Apollo 14, $R = 1.82$ for 88:0:12 mix.
- Type 1 ~ Apollo 16 highland, Type 2 ~ Apollo 14 KREEP, Type 3 ~ Apollo 11 high Ti

<table>
<thead>
<tr>
<th>Apollo Target</th>
<th>11</th>
<th>12</th>
<th>14</th>
<th>15</th>
<th>16</th>
<th>17</th>
</tr>
</thead>
<tbody>
<tr>
<td>R min</td>
<td>2.91</td>
<td>2.50</td>
<td>1.82</td>
<td>2.63</td>
<td>4.03</td>
<td>2.56</td>
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<tr>
<td>Basalt, %</td>
<td>81</td>
<td>83</td>
<td>88</td>
<td>81</td>
<td>100</td>
<td>82</td>
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<tr>
<td>Ilmenite, %</td>
<td>8</td>
<td>1</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>3</td>
</tr>
<tr>
<td>Olivine, %</td>
<td>11</td>
<td>16</td>
<td>12</td>
<td>19</td>
<td>0</td>
<td>15</td>
</tr>
<tr>
<td>Simulant Type</td>
<td>3</td>
<td>2</td>
<td>19</td>
<td>0</td>
<td>15</td>
<td></td>
</tr>
</tbody>
</table>

Data from Shigeru Aoki, Lunar Exploration Technology Office, Japan Aerospace Exploration Agency, and Hiroshi Kanamori, Institute of Technology, Shimizu Corporation
Lunar Mare Basaltic Simulants: Japanese Simulants FJS-1, Type 1-3 Root-type

<table>
<thead>
<tr>
<th>Oxide</th>
<th>FJS-1</th>
<th>Type 1</th>
<th>Type 2</th>
<th>Type 3</th>
</tr>
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<tbody>
<tr>
<td>SiO$_2$</td>
<td>49.14</td>
<td>49.1</td>
<td>49.7</td>
<td>46.0</td>
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<tr>
<td>TiO$_2$</td>
<td>1.91</td>
<td>1.9</td>
<td>1.7</td>
<td>6.7</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>16.23</td>
<td>16.2</td>
<td>14.8</td>
<td>13.7</td>
</tr>
<tr>
<td>Cr$_2$O$_3$</td>
<td>0.00</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>4.77</td>
<td>4.8</td>
<td>4.7</td>
<td>5.9</td>
</tr>
<tr>
<td>FeO</td>
<td>8.30</td>
<td>8.3</td>
<td>8.2</td>
<td>7.9</td>
</tr>
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<td>MnO</td>
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<td>0.19</td>
<td>0.19</td>
<td>0.28</td>
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<td>9.1</td>
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<td>7.8</td>
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<td>0.44</td>
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<td>LOI</td>
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<td>0.43</td>
<td>0.47</td>
<td>0.58</td>
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<td>Total</td>
<td>98.14</td>
<td>98.1</td>
<td>100.2</td>
<td>100.0</td>
</tr>
</tbody>
</table>

Simulant Materials:
- FJS-1: Mt. Fuji basalt, Japan, ~ Apollo 16
- Type 1: ~ Apollo 16
- Type 2: ~ Apollo 14
- Type 3: ~ Apollo 11
- Mixtures of Mt. Fuji basalt, ilmenite, olivine

Data from:
- Shigeru Aoki
- Lunar Exploration Technology Office, Japan Aerospace Exploration Agency
- Hiroshi Kanamori
- Institute of Technology, Shimizu Corporation
Synthetic Production of Agglutinate-like Glass Microspheres

• Weiblen: ISSP (right)
  In-flight Sustained Shockwave Plasma reactor
  MLS-1 source, plasma melting, spherical agglutinate-like
  Further work was to generate metallic Fe, other simulants

• NIST: K411 microspheres
  Plasma torch melted NIST K411 glass (MgSiCaFe glass)
  NIST SRM 2006 is K411 in 2 – 40 μm microsphere
  Particle analysis via EPMA/EDS, good agreement between
  K411 (SRM 470 bulk) and SRM 2006 microspheres

• Corning:
  Long history of research glass technology
  Initial discussions for microsphere and glass fiber research
  Small scale vs. large scale
Quality Control and Standard Development

Bulk chemistry vs. variability of MLS-1
Bulk Chemistry of MLS-1
Variability of Sub Samples: Major elements

<table>
<thead>
<tr>
<th>Oxide</th>
<th>MLS-1</th>
<th>Min</th>
<th>Max</th>
<th>Range rel to Accepted, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>43.86</td>
<td>41.7</td>
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</tr>
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<td>7.43</td>
<td>41.4</td>
</tr>
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<td>Al₂O₃</td>
<td>13.68</td>
<td>11.76</td>
<td>15.60</td>
<td>28.0</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>2.60</td>
<td>0.90</td>
<td>4.10</td>
<td>123.1</td>
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<tr>
<td>FeO</td>
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<td>MgO</td>
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<td>5.57</td>
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<td>CaO</td>
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<td>9.04</td>
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<td>Na₂O</td>
<td>2.12</td>
<td>1.97</td>
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<td>K₂O</td>
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<td>215</td>
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<td>Total</td>
<td>99.47</td>
<td>88.13</td>
<td>110.64</td>
<td>22.6</td>
</tr>
</tbody>
</table>

Gross analytical errors and/or inadequate sampling of MLS-1 source material
Bulk Chemistry of MLS-1
Variability of Sub Samples: Trace elements

<table>
<thead>
<tr>
<th>Element</th>
<th>MLS-1 ppm</th>
<th>Min ppm</th>
<th>Max ppm</th>
<th>Range rel to Average, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Th</td>
<td>14</td>
<td>13</td>
<td>15</td>
<td>14%</td>
</tr>
<tr>
<td>Sr</td>
<td>212</td>
<td>173</td>
<td>253</td>
<td>38%</td>
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<tr>
<td>Co</td>
<td>64</td>
<td>53</td>
<td>84</td>
<td>48%</td>
</tr>
<tr>
<td>Ba</td>
<td>95</td>
<td>62</td>
<td>117</td>
<td>58%</td>
</tr>
<tr>
<td>V</td>
<td>761</td>
<td>506</td>
<td>952</td>
<td>59%</td>
</tr>
<tr>
<td>Cu</td>
<td>445</td>
<td>214</td>
<td>706</td>
<td>111%</td>
</tr>
<tr>
<td>Ni</td>
<td>97</td>
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<td>113%</td>
</tr>
<tr>
<td>Cr</td>
<td>173</td>
<td>89</td>
<td>366</td>
<td>160%</td>
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<td>Zr</td>
<td>47</td>
<td>19</td>
<td>113</td>
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<td>10</td>
<td>225%</td>
</tr>
</tbody>
</table>

Variations in oxide, sulfide, others in coarse ground material, small sampling
Ranked by range error relative to average value
Oxygen Fugacity Control
Mössbauer Characterization

Oxygen fugacity of lunar vs. terrestrial igneous rocks
Mössbauer characterization / Fe valence state
Oxygen Fugacity Regimes for Lunar vs. Terrestrial Igneous Rocks

Terrestrial rocks contain Fe$^{2+}$ and Fe$^{3+}$ and form at approximately the FMQ Fe$_2$SiO$_4$ | Fe$_3$O$_4$ + SiO$_2$ buffer.

Lunar rocks contain Fe$^0$ and Fe$^{2+}$ and formed at approximately the FeWu, i.e. Fe | FeO buffer.

These buffer curves quantitatively describe the required oxygen fugacity $f_{O_2}$ to be used on processed materials.

Lunar conditions can be simulated on Earth using $f_{O_2}$ constrained systems.

Experiments / procedures on Earth must address $f_{O_2}$ control if aiming to duplicate lunar surface conditions.
Mössbauer Spectroscopy:
Confirmation of reduction Fe$^{2+}$ to Fe$^{0}$

Source Localities for Anorthosite Simulant

Stillwater intrusion, Montana, banded series
Duluth complex, Minnesota
Trends of Coexisting MgFe Minerals vs. Plagioclase, Compare Pristine Lunar vs. Stillwater

Pristine Lunar Highlands Trends
From “A comparison of fractionation trends in the lunar crust and the Stillwater Complex, L.D. Raedeke and I.S. McCallum

Stillwater Trends
From “The Stillwater Complex: A review of the geology”, I. S. McCallum
Simulant Sources for Highlands Anorthosite

• Lunar anorthosite plagioclase is Ca-rich, \(~ \text{An}_{85-96}\)
• Terrestrial anorthosites generally more Na-rich

**Stillwater intrusion**, Montana
Anorthosite, olivine-gabbro, norite; Anorthosite of MBZ possible simulant?
  Anorthosite: Plagioclase \(\text{An}_{75-80}\), Olivine \(~ \text{Fo}_{65-78}\), Augite, Fe-Ti oxides
  Active mining by Stillwater Mining Company, interested in simulant issues

**Duluth complex**, Minnesota
Anorthosite: Plagioclase \(\text{An}_{55-65}\), Olivine \(\text{Fo}_{40-66}\), Augite, Fe-Ti oxides
Troctolite: Plagioclase \(\text{An}_{52-71}\), Olivine \(\text{Fo}_{49-66}\)

Access to material in quarry, tbd
Stillwater Intrusion
Banded Series

Middle Banded Series has anorthosites with consistent plagioclase composition and relatively consistent olivine and pyroxene compositions.

Olivine Fo%  Opx En%  Plag An%
Conclusions

- Development of LRSM requires physical and chemical, mineralogical characterization via bulk and microanalytical methods
- Lunar regolith simulants JSC-1 and MLS-1 provide good framework for general LRSM discussion
- Root simulant development strategies can be quantified
- Quality control is central to LRSM standard development
- Oxygen fugacity control and Mössbauer characterization are important for processing using LRSM
- We should pursue development of anorthosite simulants at localities discussed
Additional Material
Characterization Methods
Characterization Methods: Bulk Chemistry/Structure
Non-destructive for Lunar Materials

• **X-ray fluorescence**
  New XRF systems ~10’s µm beam, microanalysis of ng to pg equivalent.
  Sensitivity major, minor, trace (µg/g)
  Can analyze insulating materials in-situ, proven for MER, on-line monitoring
  Rapid acquisition via energy-dispersive detection systems—if bright source

• **INAA Instrumental Neutron Activation Analysis**
  Analysis of mg quantity for major, minor, trace (µg/g, ng/g?)
  Extensively used for lunar trace element data
  Nuclear reactor, multiple counting experiments, not real-time

• **ICPMS**
  High analytical sensitivity, to ng/g levels
  Requires sample digestion, not real-time
MLS-1 Grain Mount
BSE Images of Unpolished Grains

Left: BSE of Ca-pyroxene with exsolution lamellae.
Right: BSE of Magnetite, Plagioclase, and Ilmenite grains. Scale 50 μm.
MLS-1 EDS Spectra of Mineral Components

BSE and X-ray maps are used to identify minerals and produce phase map
Characterization Methods: Powder X-ray Diffraction

• Mineral identification, crystallographic structure, sample mg quantity
• Modal analysis (volume percentages), detection limit ~ 0.1% X%
• Rietveld refinement: Whole pattern fitting vs. individual peak analysis, yields mineral abundance and cell information
• High temperature furnace attachment
• Lunar simulant characterization needs:
Analysis of grain size fractions: mineralogy, particle size analysis
Sintering and melting, phase/structural changes
Onset of reaction analysis
Powder X-ray Diffraction: JSC-1

Powder XRD JSC-1, minerals present at room T run:
Plagioclase (disordered), Augite, Olivine, Ilmenite, Cristobalite?
MSFC Rigaku with high-temperature stage
Data from Wilson, Carpenter
Mössbauer Spectroscopy

- A nuclear gamma resonance technique
- Several dozen nuclear isotopes exhibit the Mössbauer effect
- For planetary applications (Moon, Mars, etc.), the Fe-57 isotope is most important. The gamma source is Co-57 (270-day half life).
- Iron-containing minerals in a sample are characterized; those without iron are invisible to the technique
- Two in-situ planetary instruments are now operating on Mars
- The source is vibrated. Energy of emitted gammas are then Doppler shifted to sweep through resonant transition energies of a sample
- Spectra are superpositions of doublets and/or sextets
- Ferrous and ferric states are distinguished, e.g. olivine and jarosite
- Magnetic materials are identified, e.g. iron metal, magnetite, hematite
- Suitable for characterizing simulant material or monitoring processes
Comparison of transmission and backscatter Mössbauer geometries
Examples of Mössbauer characterization of unprocessed and processed Martian simulant [2003, unpublished]

Before processing, the spectrum is dominated by a strong central ferric doublet and weaker iron-oxide sextet in the baseline. After processing (microwave heating), there is clear evidence of reduction, the principal feature being that of a ferrous glass. The weak sextet of alpha-iron is also evident.
Root Simulants
Mass Balance Modeling of Lunar Soils Using Root Simulants

• Select root simulant components and target composition in weight % format.
  • For each root component, iterate mix fraction, (i.e., basalt, ilmenite, etc.):
  • Sum oxide wt % contribution from each simulant component
    \[(\text{FeO of mix} = c_1 \times \text{FeO in basalt} + c_2 \times \text{FeO in ilmenite} + \ldots)\]
  • Calculate R, least squares, sum of differences between root mix, \(C_i\), and Apollo target, \(L_i\), for all oxides
  • Minimum R for root simulant mix is observed for best match to Apollo soil composition.
  • Fidelity of root simulant mix is function of R, several approximations may be possible depending on accuracy needed.

\[
R = \sqrt{\frac{\sum_{i=1}^{n} (L_i - C_i)^2}{n}}
\]
## Lunar Mare Basaltic Simulants: Japanese Simulants FJS-1, MKS-1, Type 1-3

<table>
<thead>
<tr>
<th>Oxide</th>
<th>FJS-1</th>
<th>MKS-1</th>
<th>Type 1</th>
<th>Type 2</th>
<th>Type 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>49.14</td>
<td>52.69</td>
<td>49.1</td>
<td>49.7</td>
<td>46.0</td>
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<tr>
<td>TiO₂</td>
<td>1.91</td>
<td>1.01</td>
<td>1.9</td>
<td>1.7</td>
<td>6.7</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>16.23</td>
<td>15.91</td>
<td>16.2</td>
<td>14.8</td>
<td>13.7</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>4.77</td>
<td>4.78</td>
<td>4.8</td>
<td>4.7</td>
<td>5.9</td>
</tr>
<tr>
<td>FeO</td>
<td>8.30</td>
<td>7.50</td>
<td>8.3</td>
<td>8.2</td>
<td>7.9</td>
</tr>
<tr>
<td>MnO</td>
<td>0.19</td>
<td>0.22</td>
<td>0.19</td>
<td>0.19</td>
<td>0.28</td>
</tr>
<tr>
<td>MgO</td>
<td>3.84</td>
<td>5.41</td>
<td>3.8</td>
<td>8.1</td>
<td>7.3</td>
</tr>
<tr>
<td>CaO</td>
<td>9.13</td>
<td>9.36</td>
<td>9.1</td>
<td>8.4</td>
<td>7.8</td>
</tr>
<tr>
<td>Na₂O</td>
<td>2.75</td>
<td>1.90</td>
<td>2.8</td>
<td>2.6</td>
<td>2.6</td>
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<tr>
<td>K₂O</td>
<td>1.01</td>
<td>0.58</td>
<td>1.0</td>
<td>0.92</td>
<td>0.87</td>
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<tr>
<td>P₂O₅</td>
<td>0.44</td>
<td>0.14</td>
<td>0.44</td>
<td>0.40</td>
<td>0.39</td>
</tr>
<tr>
<td>LOI</td>
<td>0.43</td>
<td>0.50</td>
<td>0.43</td>
<td>0.47</td>
<td>0.58</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>98.14</strong></td>
<td><strong>100.0</strong></td>
<td><strong>98.1</strong></td>
<td><strong>100.2</strong></td>
<td><strong>100.0</strong></td>
</tr>
</tbody>
</table>

**Simulant Materials:**
- **FJS-1:** Mt. Fuji basalt, Japan
- **MKS-1:** Type 1, 2, 3: Physical mixtures of Mt. Fuji basalt, ilmenite, olivine

Data from:
- Shigeru Aoki
  Lunar Exploration Technology Office, Japan Aerospace Exploration Agency
- Hiroshi Kanamori
  Institute of Technology, Shimizu Corporation
Martian Soil Simulant Mars-1
Mars Rock and Soil Chemistry / Geology

• Martian meteorites (>29 found to date – 7/2004 New Nakhlite! MIL-03346)
  SNC, ALH84001: peridotites, pyroxenites, basaltic rocks (ol, cpx, opx, plag)
• Viking 1 and Viking 2 landers (1976)
  XRFS, GCMS, NMS, Magnetic: soil chemistry, organics, magnetic minerals
• Mars Global Surveyor
  Global imaging calibrated using lander and rover data
• Pathfinder (1997), Spirit, Opportunity (2004) – Hematite (Fe₂O₃) Identified
  Panoramic Camera: High resolution imaging
  Thermal Emission Spectrometer: Mineral identification, test for H₂O, OH
  Mossbauer Spectrometer (Co⁵⁷ source): Fe oxidation state
  APXS (Cm²⁴⁴) Alpha Proton X-ray Spectrometer: Major element rock analysis
  Microscopic Imager: Fine scale imaging of grains
JSC Mars-1 Martian Soil Simulant: Typical Grain

- Rock fragment with glass, plagioclase, augite, Ti-magnetite, palagonite alteration.
JSC Mars-1 Compared to Martian Soil / Rock

- Mars-1 Simulant
  XRF norm volatile free
  Fe$_2$O$_3$* 15.6
  Fe$^{3+}$/Fe$^{2+}$ ~ 3.2
- Pathfinder Soil
  APXS A-2 Deploy
- Pathfinder SFR
  Soil-free rock
- Viking Lander 1
  XRF norm volatile free
  (Total 89%), avg 3, Fe$_2$O$_3$ *
- Shergotty meteorite
  INAA
- Yellow: Mars-1 differs from Soil / SFR
- Blue: Soil differs from SFR

<table>
<thead>
<tr>
<th></th>
<th>Mars-1</th>
<th>Soil</th>
<th>SFR</th>
<th>VL-1</th>
<th>Sher</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO$_2$</td>
<td>43.5</td>
<td>40.9</td>
<td>57.7 ± 1.5</td>
<td>48.4</td>
<td>51.36</td>
</tr>
<tr>
<td>TiO$_2$</td>
<td>3.8</td>
<td>0.7</td>
<td>0.5 ± 0.15</td>
<td>0.74</td>
<td>0.87</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>23.3</td>
<td>10.4</td>
<td>12.3 ± 0.7</td>
<td>8.2</td>
<td>7.06</td>
</tr>
<tr>
<td>Cr$_2$O$_3$</td>
<td>0.3</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>15.6</td>
<td>21.2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FeO</td>
<td></td>
<td></td>
<td>14.2 ± 0.8</td>
<td></td>
<td>19.41</td>
</tr>
<tr>
<td>MnO</td>
<td>0.3</td>
<td>0.5</td>
<td></td>
<td></td>
<td>0.52</td>
</tr>
<tr>
<td>MgO</td>
<td>3.4</td>
<td>8.7</td>
<td>0.8 ± 0.8</td>
<td>6.7</td>
<td>9.28</td>
</tr>
<tr>
<td>CaO</td>
<td>6.2</td>
<td>6.1</td>
<td>6.7 ± 0.5</td>
<td>6.6</td>
<td>10</td>
</tr>
<tr>
<td>Na$_2$O</td>
<td>2.4</td>
<td>3.2</td>
<td>4.2 ± 0.6</td>
<td></td>
<td>1.29</td>
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<tr>
<td>K$_2$O</td>
<td>0.6</td>
<td>0.5</td>
<td>1.2 ± 0.08</td>
<td>&lt; 0.17</td>
<td>0.16</td>
</tr>
<tr>
<td>P$_2$O$_5$</td>
<td>0.9</td>
<td>0.9</td>
<td>0.4 ± 0.2</td>
<td></td>
<td>0.8</td>
</tr>
<tr>
<td>SO$_3$</td>
<td></td>
<td></td>
<td>6.0 ± 0.04 S</td>
<td>7.4</td>
<td>0.13 S</td>
</tr>
<tr>
<td>Cl</td>
<td></td>
<td>0.7</td>
<td>0.4 ± 0.1</td>
<td>0.8</td>
<td>0.01</td>
</tr>
<tr>
<td>LOI</td>
<td>(22)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>(100)</td>
<td>100.1</td>
<td>98.5</td>
<td>(100)</td>
<td>100.9</td>
</tr>
</tbody>
</table>
JSC Mars-1 BSE Image
Range of chemical weathering