THE 2010 FIELD DEMONSTRATION OF THE SOLAR CARBOTHERMAL REDUCTION OF REGOLITH TO PRODUCE OXYGEN

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First Joint Meeting of the
Space Resources Roundtable and the
Planetary & Terrestrial Mining Sciences Symposium
Colorado School of Mines, Golden, Colorado
8 - 10 June 2010

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Outline

- Introduction
- Background Information
- Results of the Field Demonstration
- Open Issues
- Future Plans
- Conclusions

Introduction

- The carbothermal reduction process is a mature terrestrial technology to produce high-purity silicon from silica (SiO_2)
- It can also be used to produce oxygen from the regolith through the carbon reduction of minerals that contain silicon, iron, and titanium oxides (such as ilmenite and silicates)
- Over 28% of the mass of JSC-1A lunar regolith simulant can potentially be extracted as oxygen, mostly from reduction of silicates
- Silicates are believed to be a significant component of the regolith on many extraterrestrial bodies
- Therefore, the carbothermal reduction process can be efficiently used with regolith from **any location** on the Moon, asteroids, Martian moons, or Mars with little or no beneficiation

Carbothermal Reduction Background

- Carbothermal reduction produces oxygen using the following reactions (MO_x = FeO, Fe_2O_3, TiO_2 [partial reduction], and SiO_2):

$$MO_x + x\,CH_4 \xrightarrow{>1600\text{ C}} M + x\,CO + 2x\,H_2 \qquad (1)$$

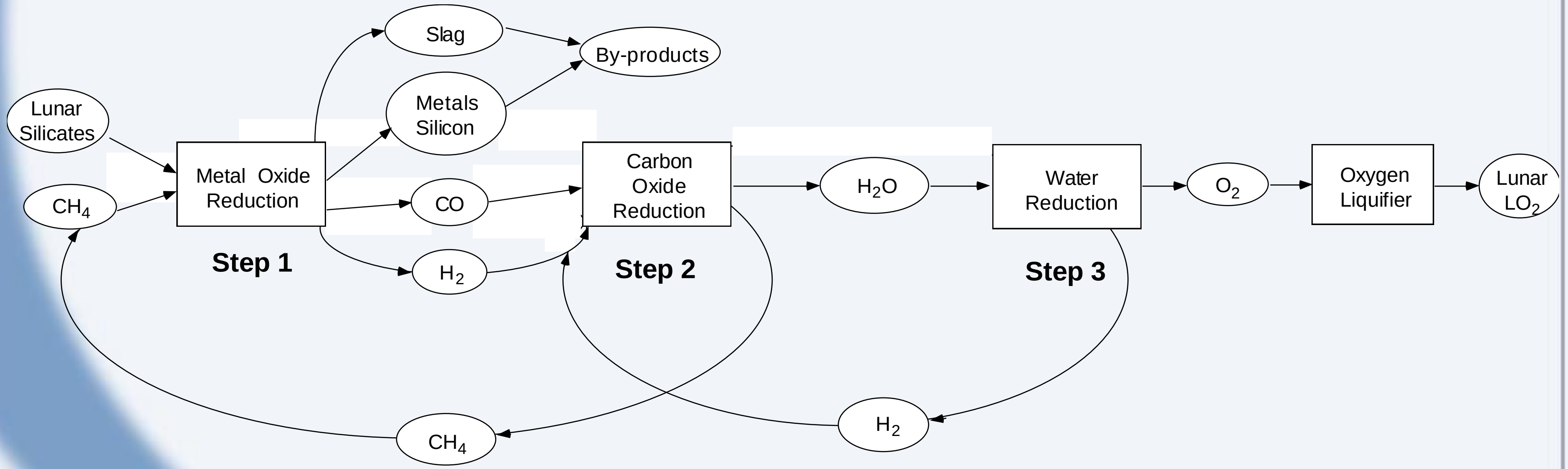
$$x\,CO + 3x\,H_2 \longrightarrow x\,CH_4 + x\,H_2O \qquad (2)$$

$$x\,H_2O \longrightarrow x\,H_2 + 0.5x\,O_2 \qquad (3)$$

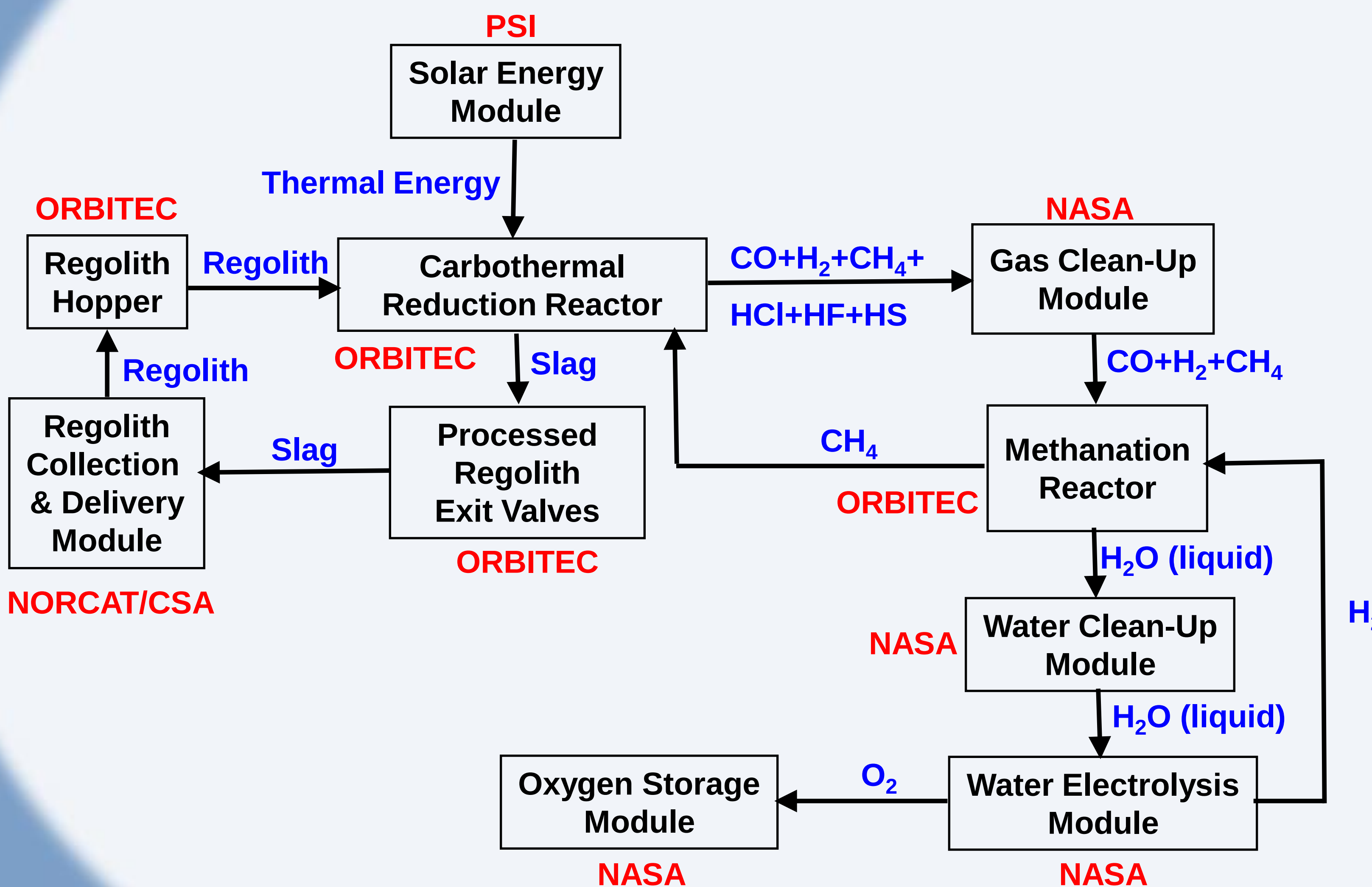
$$MO_x \longrightarrow M + 0.5x\,O_2 \qquad (4)$$

Carbothermal Reduction Background

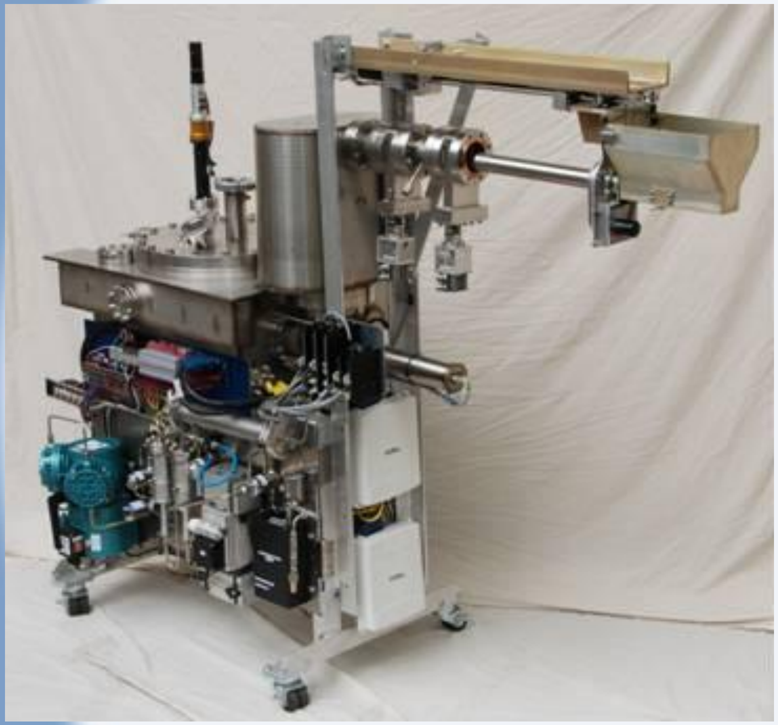
- The baseline carbothermal reduction process has three basic steps
  - Step 1. Carbon Reduction of Metallic Oxides
  - Step 2. Methane Reformation
  - Step 3. Water Electrolysis



Carbothermal Regolith Processing System



Carbothermal Regolith Reduction Module



Carbothermal Regolith Reduction Module

- ORBITEC developed the Carbothermal Regolith Reduction Module to demonstrate the extraction of oxygen from lunar regolith simulant using concentrated solar energy
  - Automated filling of the regolith hopper and transfer to carbothermal reduction reactor
  - Automated gas handling system, including gas clean-up beds and methanation reactor
  - Automated removal of processed regolith
  - Ability to operate remotely through an http interface
- Hardware is sized to produce 1 MT O_2 per year (with seven melts)

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Solar Energy Collection and Delivery Module

Solar Energy Collection and Delivery Module integrated with the Carbothermal Regolith Reduction Module

- Physical Sciences Inc. built a Solar Energy Collection and Delivery Module that provides concentrated solar energy to the Carbothermal Regolith Reduction Module
- Seven solar concentrators are mounted on an array with two-axis tracking of the sun
- The solar energy from each concentrator is delivered to the carbothermal reactor via a fiber optic cable
- Each fiber optic cable delivers up to 100 W (total of 600 to 700 W) of concentrated solar energy

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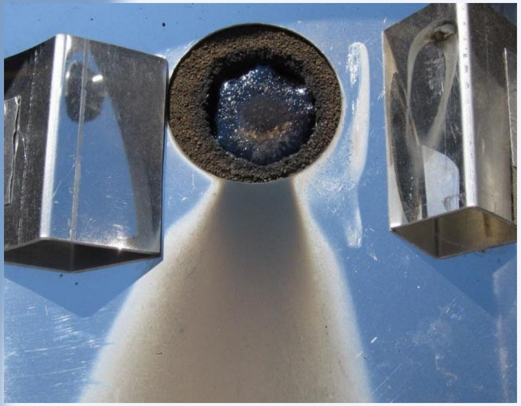
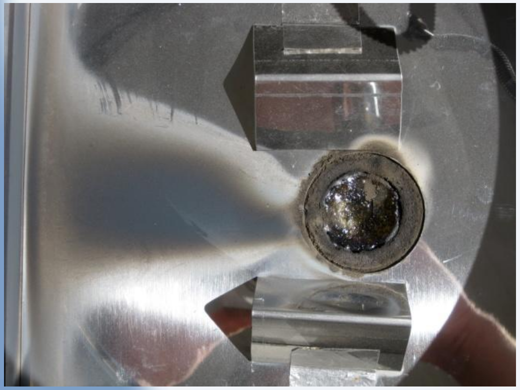
Carbon Reduction with Solar Energy



Processed Tephra



Processed JSC-1A Simulant

- Testing at ORBITEC demonstrated oxygen yields up to 10.3%wt with processing times up to 80 minutes for JSC-1A lunar regolith simulant and Hawaiian tephra
- The carbothermal reduction process produces silica fume as an intermediate product, so keeping the end of the quartz rod clean has been a challenge
- ORBITEC has built and successfully tested a gas nozzle that keeps the end of the quartz rod clean during processing (aerodynamic window)

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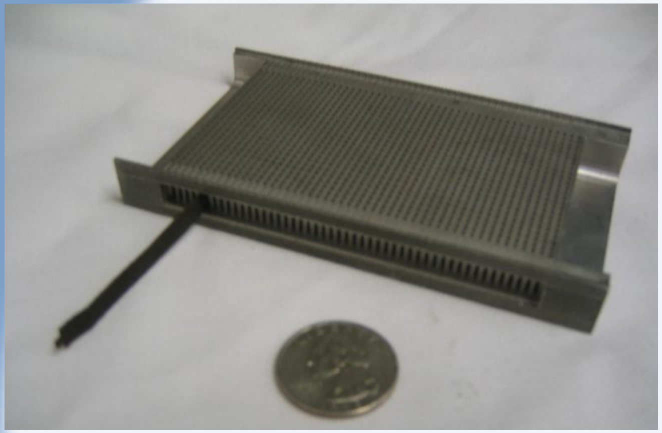
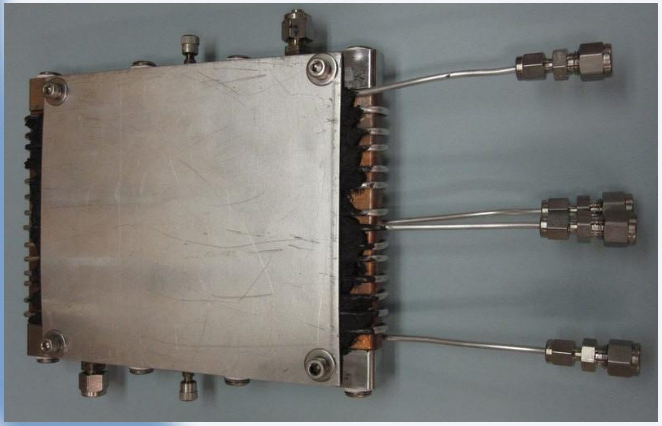
Methane Reformation Reactor



PNNL Methanation Reactor



ORBITEC Methanation Reactor

- A two-stage microchannel methanation reactor was built by Pacific Northwest National Laboratory
  - First stage operates at ~450 C to increase kinetics followed by second stage at ~350 C to achieve better CO conversion
  - Problems with internal coking limits incoming H_2:CO ratio to 3.7:1 or higher
  - Complete CO conversion with 4.4% CO_2 and 51.3% H_2 in exit stream
- The ORBITEC methanation reactor uses an industrial nickel-based catalyst
  - Operates at 250 C = slower kinetics but better CO/CO_2 conversion
  - Can be operated with incoming H_2:CO ratio to 3:1 (stoichiometric) with no coking
  - Complete conversion of CO with 0.7% CO_2 and 13.6% H_2 in exit stream

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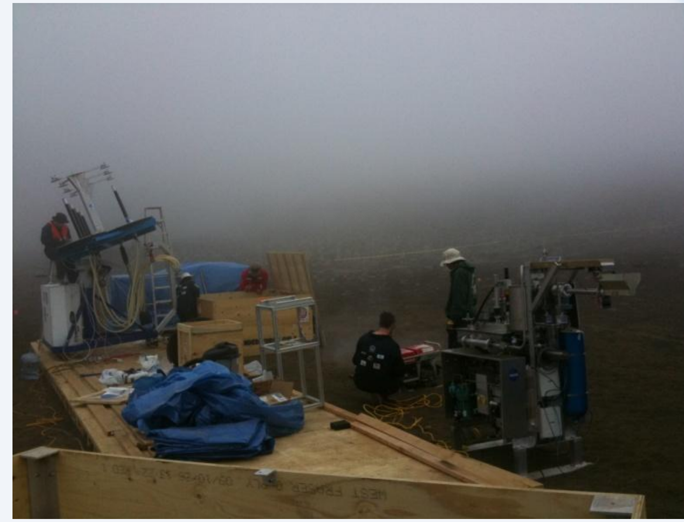
Field Demonstration

- The NASA ISRU program is raising the TRL of various technologies through analog field demonstrations, e.g. Nov. 2008, Jan./Feb. 2010
- The Carbothermal Processing System (CT System) was selected to be demonstrated as part of the 2010 International Lunar Surface Operations and ISRU Analog Test
- The field demonstration was held January 24 - February 14, 2010 at an analog test site on Mauna Kea on the Big Island of Hawaii
- The analog test site is located at an elevation of ~9,000 feet (2,743 m) near the Visitor Center and Hale Pohaku which is the astronomer’s dormitory

I came to Hawaii for this?????



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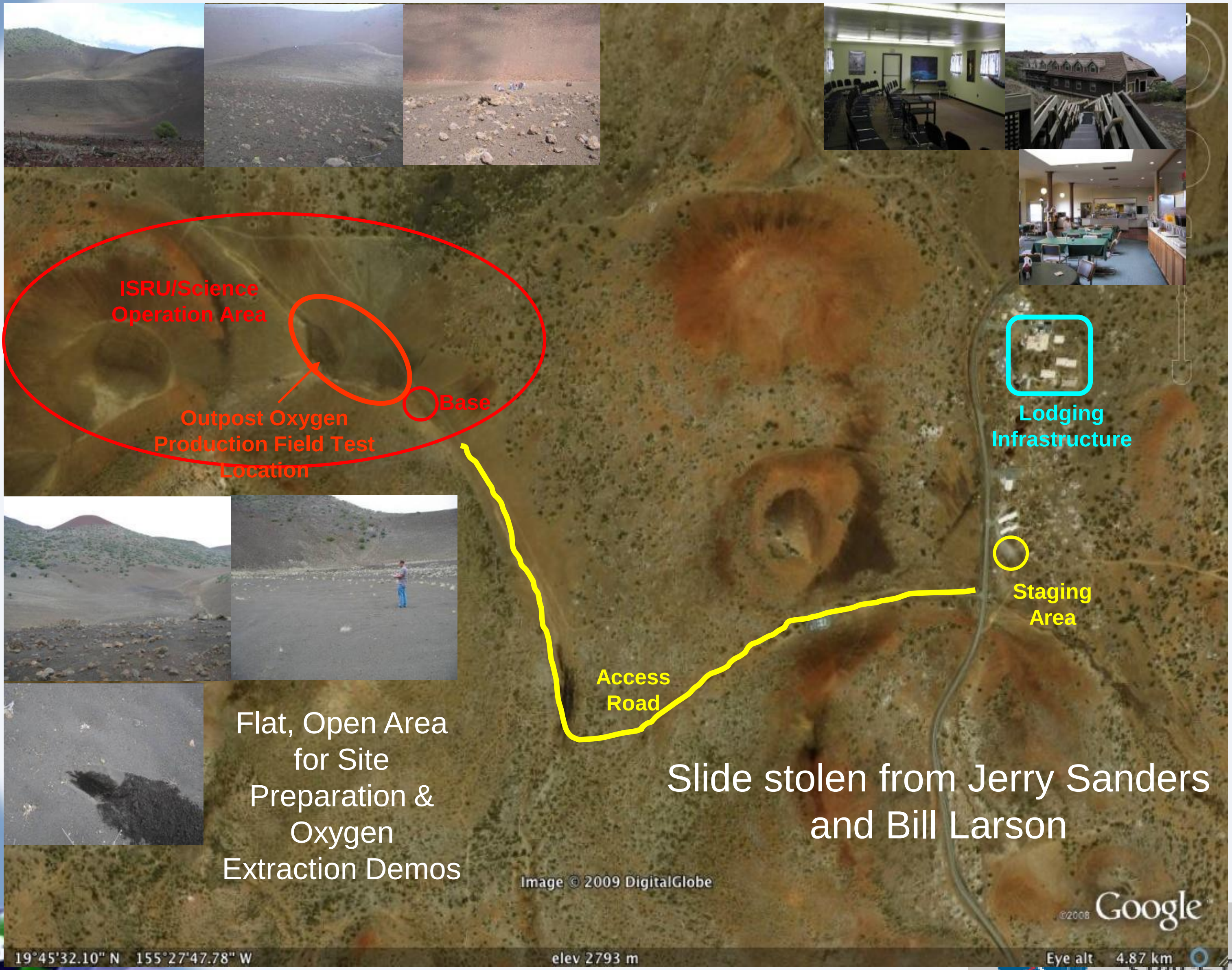
Analog Test Site on Mauna Kea



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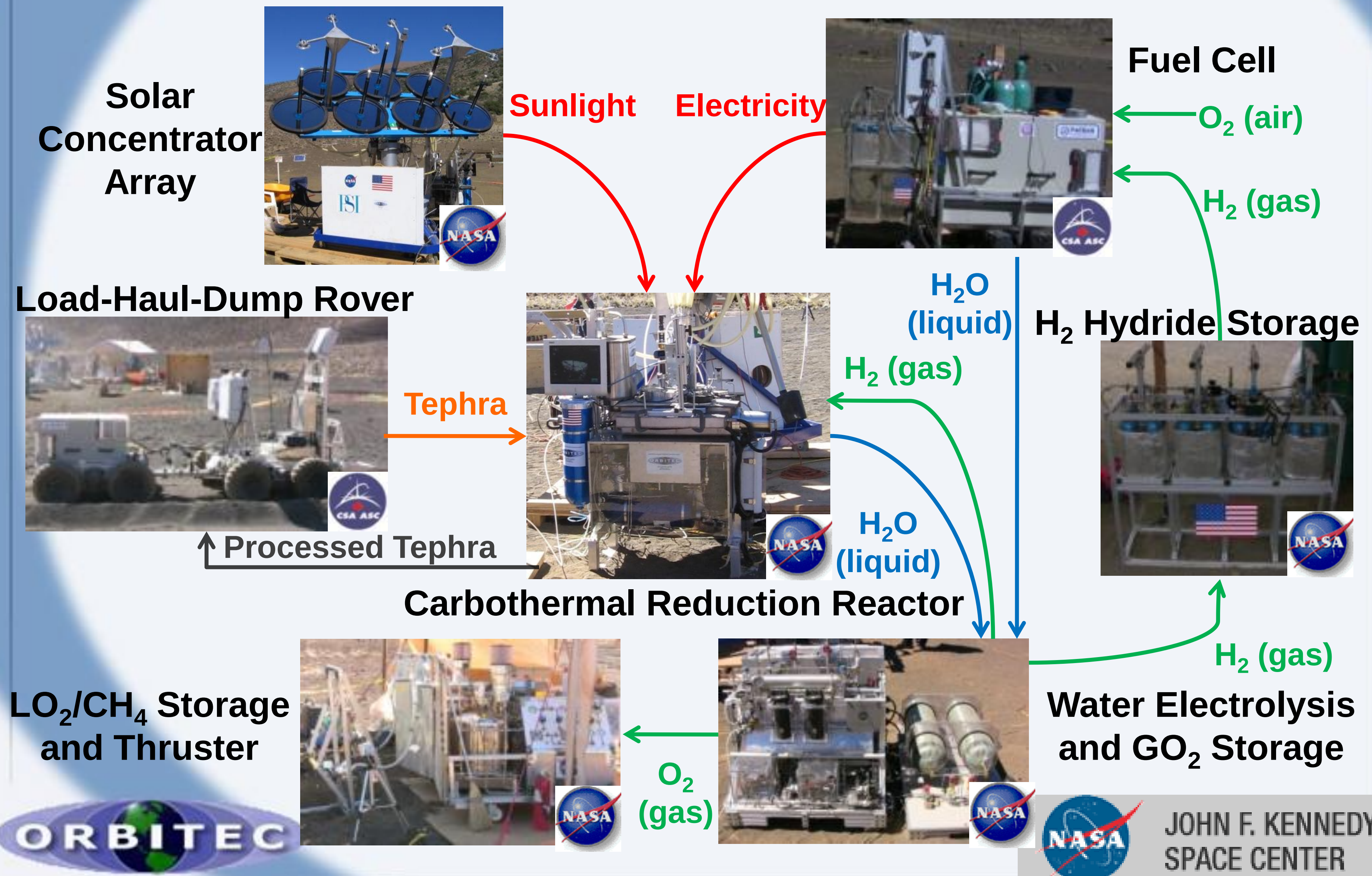
Lunar/Mars-Like Terrain on Mauna Kea

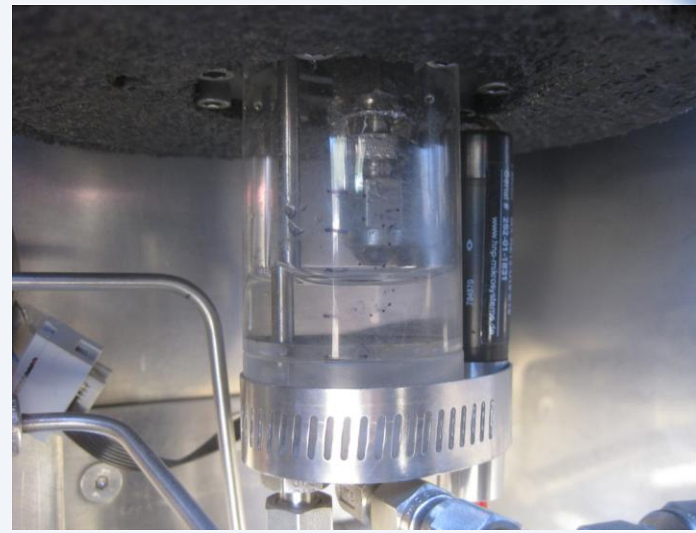
Field Demonstration Overview



Summary Video of the Field Demonstration

Successful Results

The 17 tephra melts processed in the CT System during the field demonstration



Liquid water collected in the water reservoir downstream of the condenser

Processed tephra melt from day 6 - Experiment 1

Processed tephra melts from Day 5 - Experiments 1, 2 & 3

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CT System Performance Data
(10% O_2 Total Yield & 2-8% Reduction Yield)

<table>
<tr><th>Date</th><th>Batch #</th><th>Tephra Melt Mass (g)</th><th>Processing Time (min.)</th><th>Estimated Delivered Solar Power (W)</th><th>Water Collected (g)</th><th>O_2 Yield by Mass (%) [from water]</th><th>O_2 Yield by Mass (%) [from GC]</th><th>O_2 Produced (g) [from GC]</th></tr>
<tr><td>Day 5 (2-Feb-10)</td><td>1</td><td>24.5</td><td>80</td><td>450-500</td><td rowspan="4">-</td><td rowspan="4">-</td><td rowspan="4">4.0</td><td rowspan="4">3.7</td></tr>
<tr><td>Day 5 (2-Feb-10)</td><td>2</td><td>23</td><td>80</td><td>550</td></tr>
<tr><td>Day 5 (2-Feb-10)</td><td>3</td><td>10.0</td><td>80</td><td>510→360</td></tr>
<tr><td>Day 6 (3-Feb-10)</td><td>1</td><td>32.2</td><td>90</td><td>555</td></tr>
<tr><td>Day 6 (3-Feb-10)</td><td>2</td><td>27.9</td><td>120</td><td>550→510</td><td rowspan="4">11.5</td><td rowspan="4">9.6</td><td rowspan="4">N/A</td><td rowspan="4">N/A</td></tr>
<tr><td>Day 7 (4-Feb-10)</td><td>1</td><td>24.5</td><td>80</td><td>550</td></tr>
<tr><td>Day 7 (4-Feb-10)</td><td>2</td><td>26.0</td><td>80</td><td>560-580</td></tr>
<tr><td>Day 7 (4-Feb-10)</td><td>3</td><td>17.9</td><td>80</td><td>570→540</td></tr>
<tr><td>Day 7 (4-Feb-10)</td><td>4</td><td>13.0</td><td>80</td><td>510→450</td><td rowspan="4">8.2</td><td rowspan="4">9.9</td><td>8.1</td><td>1.1</td></tr>
<tr><td>Day 8 (5-Feb-10)</td><td>1</td><td>27.6</td><td>80</td><td>570</td><td>3.7</td><td>1.1</td></tr>
<tr><td>Day 8 (5-Feb-10)</td><td>2</td><td>14.1</td><td>80</td><td>530</td><td>5.0</td><td>0.7</td></tr>
<tr><td>Day 8 (5-Feb-10)</td><td>3</td><td>11.5</td><td>80</td><td>500</td><td>5.3</td><td>0.6</td></tr>
<tr><td>Day 9 (6-Feb-10)</td><td>1</td><td>15.9</td><td>120</td><td>575</td><td rowspan="4">11.8</td><td rowspan="4">10.0</td><td>3.9</td><td>0.7</td></tr>
<tr><td>Day 10 (8-Feb-10)</td><td>1</td><td>27.9</td><td>120</td><td>500→530</td><td>N/A</td><td>N/A</td></tr>
<tr><td>Day 10 (8-Feb-10)</td><td>2</td><td>19.8</td><td>100</td><td>590→300</td><td>4.6</td><td>1.0</td></tr>
<tr><td>Day 11 (9-Feb-10)</td><td>1 & 2</td><td>30.8</td><td>160 (total)</td><td>570</td><td>2.0</td><td>0.6</td></tr>
</table>

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ORBITEC Methanation Reactor (~100% CO Conversion)

	Feed Ratio	Flow Rate	Inlet Gas Stream Composition (%mol)				Outlet Gas Stream Composition (%mol)				Conversion Rate (%)	
	(H_2):$(CO+CO_2)$	(sccm)	H_2	CH_4	CO	CO_2	H_2	CH_4	CO	CO_2	CO	CO_2
Lab	3:1	1480	19.9	0.0	5.8	0.81	1.3	5.7	0.00	0.10	100	58
	3:1	1482	37.2	0.0	11.6	0.94	2.1	13.4	0.00	0.01	100	63
Field Demonstration	2.2:1	1050	7.1	4.1	2.6	0.66	0.51	7.9	0.00	0.84	100	-27
	2.4:1	1050	8.5	5.5	3.1	0.44	0.85	9.6	0.00	0.46	100	-4
	2.8:1	1050	6.8	4.0	3.2	1.41	0.62	9.1	0.03	0.59	99	56
	3:1	1050	9.6	4.0	2.8	0.43	0.68	8.8	0.00	0.24	100	43
	5.4:1	1050	8.1	3.2	2.1	0.47	1.25	7.8	0.00	0.09	100	80
	6.6:1	1050	7.3	4.2	1.5	0.48	1.27	7.2	0.00	0.02	100	97
	7.0:1	1050	7.3	3.1	1.4	0.48	1.37	6.3	0.00	0.02	100	95

Excellent Gas Scrubber Performance

- NASA/KSC measured the concentrations of fluorine, chlorine, and sulfur compounds in the water produced in the CT System and predicted the concentrations that were present in the gas phase
- <u>Goal of reducing gas concentrations to less than 1 ppm was achieved</u>

Sample Name	Water Concentration			Predicted Gas Concentration		
	F	Cl	SO_4	F	Cl	SO_4
	mg/L	mg/L	mg/L	ppm	ppm	ppm
Carbothermal Sample 1	0.306	14.077	0.019	0.33	0.66	0.00
Carbothermal Sample 2 (deionized water)	0.365	0.085	0.118	-	-	-
Carbothermal Sample 3	0.374	16.429	0.187	0.04	0.92	0.00

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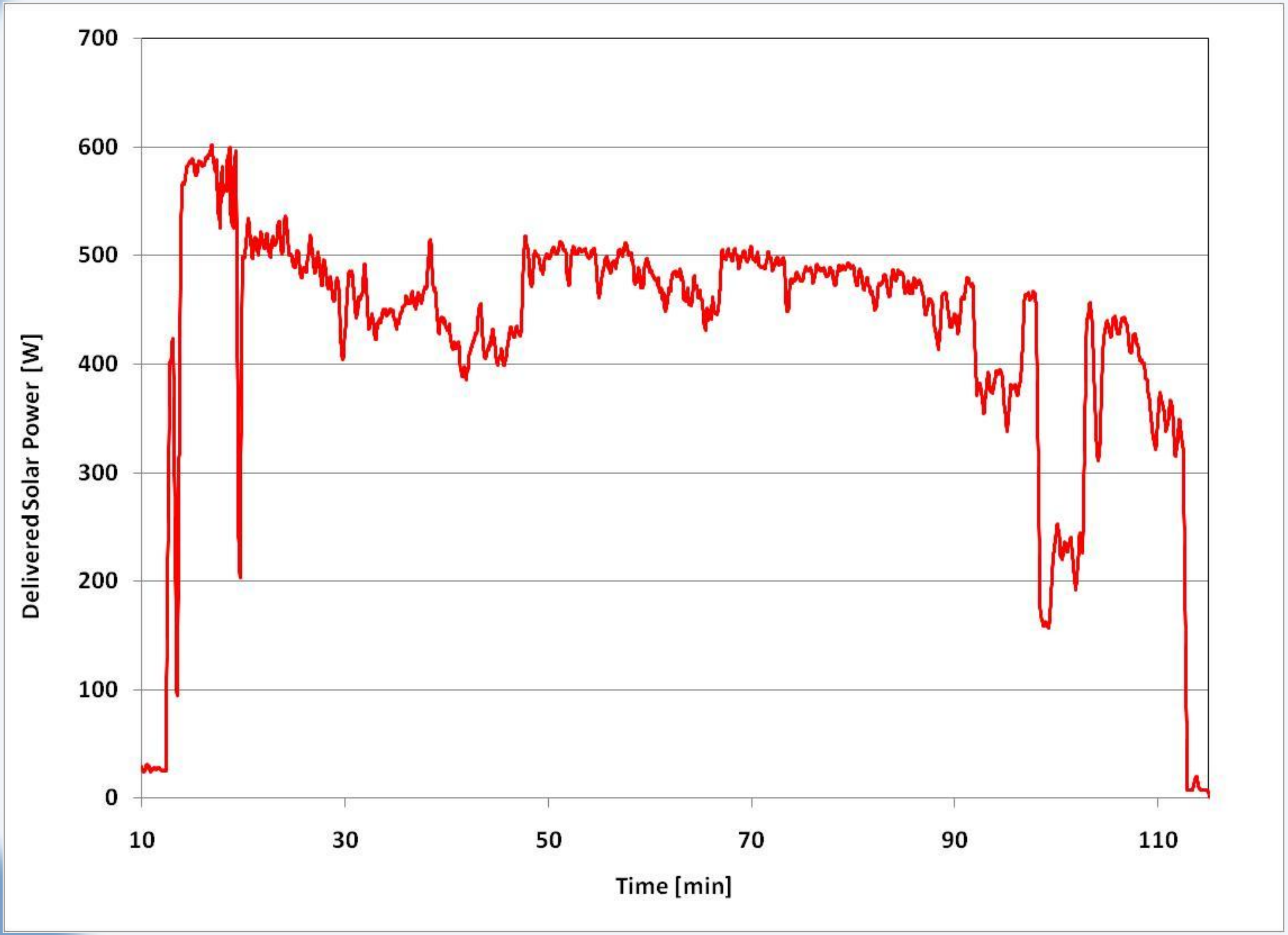
CT Chamber Seals Worked Very Well

- The overall leak rate of the Carbothermal Reduction Chamber was measured throughout the field demonstration
- The leak rate measurements verify that the regolith inlet valves and processed regolith exit valve maintained their sealing performance after multiple uses with the tephra

Date	Leak Rate (psi/min)	Leak Rate (sccm)
Day 4	0.0048	31.5
Day 8	0.0025	16.4
Day 9	0.0024	15.7
Day 10	0.0018	11.8

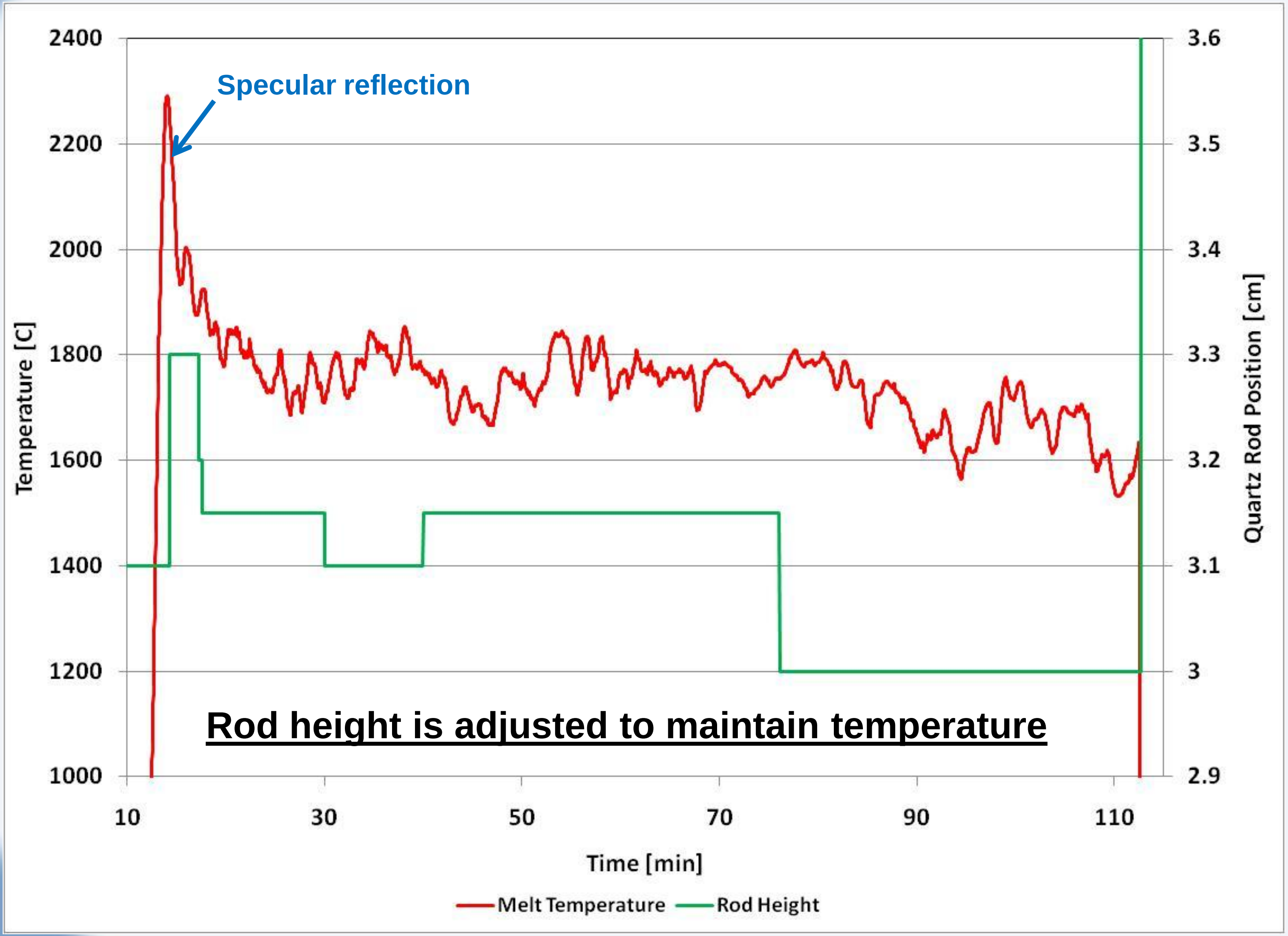
Decreased over time!

Delivered Solar Power (8-Feb-2010 Exp. 2)

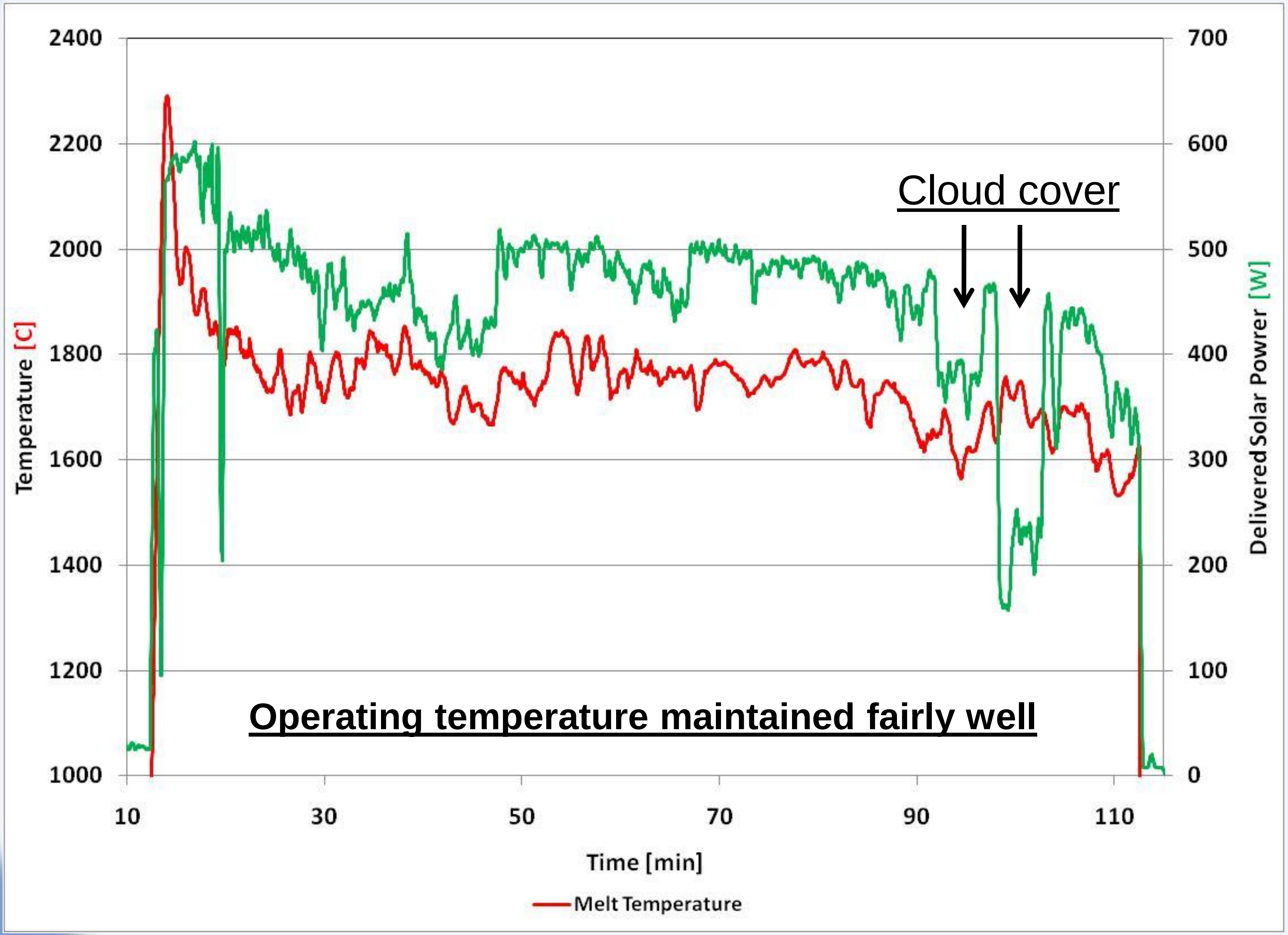


(Based on solar flux measurements)

Measured Melt Temperature (8-Feb-2010 Exp. 2)



Power vs. Temperature (8-Feb-2010 Exp. 2)



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Major Accomplishments

- Demonstrated closed-loop operation of the carbothermal reduction process using direct solar energy at an analog test site
- Integrated system operated in the field for 7 days with multiple processing batches each day
- Demonstrated oxygen yields up to 8.1% by mass from the tephra based on GC measurements and up to 10% based on the water collected
- Removed the Cl, F and S compounds evolved from the tephra to a level of less than 1 ppm (level safe for methanation reactor catalyst)
- Virtually 100% one-pass conversion efficiency of CO in the ORBITEC Methanation Reactor with a stoichiometric inlet composition
- Demonstrated remote operations from JSC
- No measurable degradation in the sealing performance of the regolith inlet and exit valves on the Carbothermal Reduction Chamber during operation at the dusty analog test site

Open Issues

- Carbon cap formation
  - Easily controlled with consistent heat source, e.g. lunar sunlight
  - Carbothermal reduction with multiple melt zones not tested yet
- Protection of quartz rod
  - Minimum quartz rod height of 3 cm required, but larger distances can be used with higher power light source
- Terrestrial operations
  - Solar flux variations (atmospheric density, humidity, clouds, dust, corrosion) have a significant impact on processing
  - Partial solution: solar power simulator that needs to be calibrated
- Comparison to alternate heat sources
  - Lasers, heat pipes, resistance heaters, and microwaves
  - Current NASA trade study and proposed contractor study
  - ORBITEC Ph.1 SBIR testing coated heating elements and crucibles

Future Project Schedule

- June – September 2010
  - Demonstrate the single-melt operation of the Carbothermal Regolith Reduction Module using up to ~1 kW of simulated solar power (using two 500 W diode lasers)
  - Compare the melt size growth rate for a single melt with three adjacent melts in open air (no reduction process) using a combination of a CO_2 laser and two 500 W diode lasers
- October 2010 – September 2011 (Proposed)
  - Complete FY10 plan to demonstrate multi-melt reductions in the Carbothermal Regolith Reduction Module
  - Develop a conceptual design for a carbothermal reduction technology demonstration unit on a robotic precursor mission
  - Perform a heat source trade study for precursor flight unit
  - Initiate studies of separation/use of solid products: iron and silicon metals, slag for construction

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Conclusions

- The Carbothermal Regolith Reduction Module successfully demonstrated the complete carbothermal regolith reduction process for the first time using concentrated solar energy in a remotely operated, semi-autonomous manner at a relevant scale at a lunar analog test site
- Overall performance of the system was excellent, although input solar energy was somewhat less than anticipated, reducing the melt sizes and oxygen yields
- Oxygen generated by the end-to-end process was included in the LOX burned in the methane engine firing, demonstrating "Dust-to-Thrust"
- ORBITEC has done an outstanding job completing this phase of the project

Acknowledgements

- This work is funded under a Phase III SBIR contract from the NASA Kennedy Space Center

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