



# Abstract #1687

## English

### Production of Commodities from Extraterrestrial Surfaces via Thermal Decomposition and Fractional Separation

In-Situ Resource Utilization (ISRU) involves mining and converting in-situ resources into products that can reduce mission mass, cost of human exploration in the moon, Mars, and asteroids. Regolith and soil of these extraterrestrial sites contain resources that can be harvested into products. The state of the art technologies for current ISRU processes are heavily dependent on terrestrial precursors, too complex, and are not designed to operate taking advantages of the extraterrestrial environment in which the resource is found, especially inertness, high-vacuum and low-gravity. This paper presents the results, to date, of a proof of concept research study on the feasibility of a sustainable thermal-driven process approach to manufacture different types of extraterrestrial commodities. This novel process approach requires none or minimal terrestrial precursors and uses extraterrestrial vacuum ambient conditions (1E-10-1E-12 torr at the moon and asteroids, 2-4 torr at Mars) as a key driven force to convert local extraterrestrial regolith soil into valuable products/commodities. Temperature, vacuum level, and process control operation determine the type of product that can be manufactured, such as a) building materials and radiation shields at moderate temperature (just below melting point), b) 3D printing feedstock and thin films at melting temperature, and c) controlled fractional decomposition and generation of oxygen, metals, and alloys at temperature above melting point.

## French

### No abstract title in French

No French resume

## Author(s) and Co-Author(s)

Mr. Jesus Adolfo Dominguez  
(UnknownTitle)  
Florida Institute of Technology

Dr. Jonathan Whitlow  
(UnknownTitle)  
Florida Institute of Technology

Mr. Jonathan Elm  
(UnknownTitle)  
Florida Institute of Technology



# Profile of Mr. Jesus Dominguez

## General

Email(s): [jesus.a.dominguez@nasa.gov](mailto:jesus.a.dominguez@nasa.gov)

Position:

Preferred Language: [Language not defined]

## Addresses

Business

Home

## Biographies

### Biography submitted with the abstract

PhD Candidate at Florida Institute of Technology (Chemical Engineering Department) holding Master Degrees in Chemical Engineering and Computer Science. Multidisciplinary research engineer with solid knowledge and vast experience in: multi-physics modeling and simulation, soft-computing-based development of intelligent systems and image analysis algorithms, scientific algorithm/software development, and exploratory chemical process implementation. Expert in solving research and development problems that require skills, experience, and academic background in chemical engineering and computer science. Author and co-author of NASA patents licensed to several companies for further development and potential commercialization. Also author and co-author of peer-reviewed publications and presentations. Recipient of various NASA awards including 2014 Silver Achievement Medal for project support contribution to Space Center Innovation Fund (CIF). Other key recognized achievements at NASA include: KSC 2004 Software of the year, NASA Innovation on Advanced Concepts (NIAC) project participation, and image analysis work during the Orbiter Columbia accident investigation.

### Biography in the user profile

## Collaborators

### Author(s) and Presenter(s)

Author(s):

Mr. Jesus Adolfo Dominguez  
[Unknown Title]  
Florida Institute of Technology

Dr. Jonathan Whitlow  
[Unknown Title]  
Florida Institute of Technology

Mr. Jonathan Elm  
[Unknown Title]  
Florida Institute of Technology

---

Presenter(s):

Mr. Jesus Adolfo Dominguez  
[Unknown Title]  
Florida Institute of Technology

# Production of Commodities from Extraterrestrial Surfaces via Thermal Decomposition and Fractional Separation

Jesus A. Dominguez, Jonathan Whitlow, Jonathan Elm

Florida Institute of Technology  
Melbourne, Florida

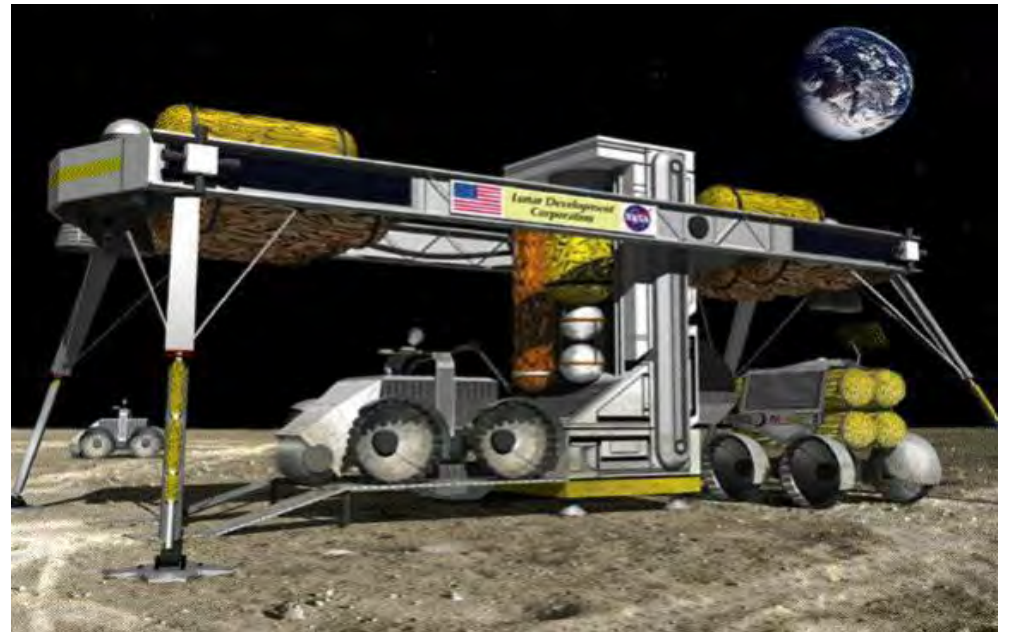


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- In-Situ Resource Utilization (ISRU) involves mining and converting in-situ resources into products that can reduce mission mass, cost, and/or risk of human exploration in the moon, Mars (and its moons), and near earth asteroids.
- Regolith and soil of these extraterrestrial sites contain resources that can be harvested into products, water, oxygen and metals .





The state of the art technologies for many current ISRU processes either do not exist or are

- **heavily dependent** on terrestrial precursors,
- complex,
- inefficient (mass, power, and/or volume),
- not designed to operate taking advantages of the extraterrestrial environment in which the resource is found, especially **high-vacuum** and **low-gravity**.



A key part of our research work is to be able to:

- demonstrate experimentally the feasibility of using a **terrestrial-precursor-free** thermal-driven-based process to transform extraterrestrial in-situ resources into useable products.
- estimate the temperature distribution and heat flux throughout the raw material at which it becomes product as temperature cannot be directly measured as the raw sample is housed within double crucible inside the **Vacuum Fractional Thermal Column (VFTC)** that is completely sealed to hold vacuum conditions.
- Experimentally evaluate **gradient temperature** (bulk scale) effect on melt transport profile.

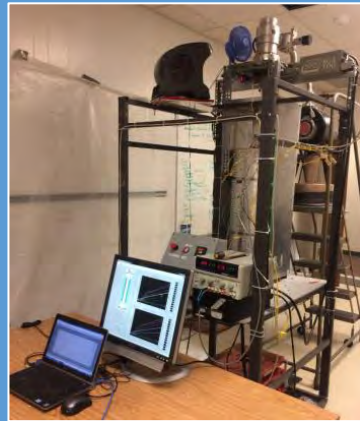
Evaluate the feasibility of using a single **thermal-driven** process under **high vacuum** with **none or minimal terrestrial precursors** to:

1) convert extraterrestrial regolith/soil into:

- Building materials.
- Coating & 3D printing feedstock.
- Gaseous O<sub>2</sub> & metals/alloys.

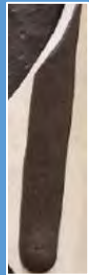
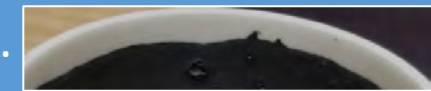
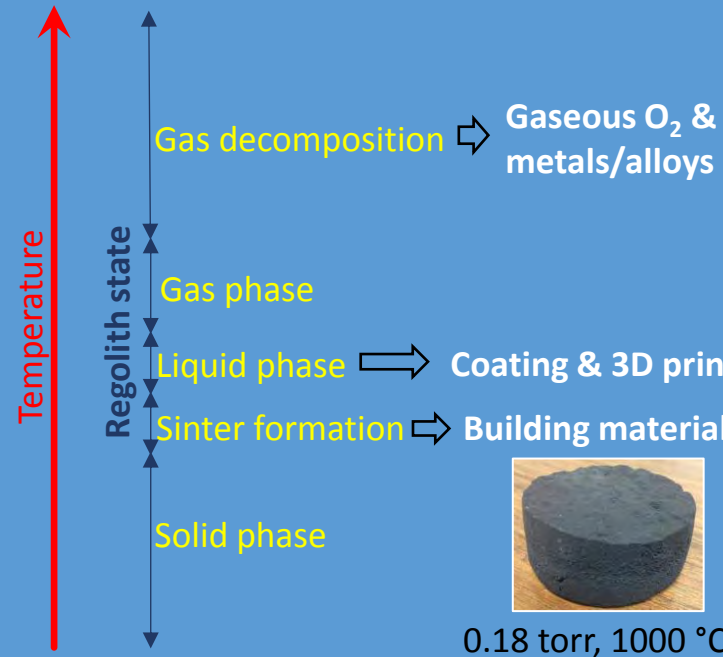
2) be key part of future mining process in asteroids, moons, and planets to **extract and refine** simultaneously valuable metals/allows by decomposing their ores and releasing surplus constitutes (O<sub>2</sub> forming metal oxides).





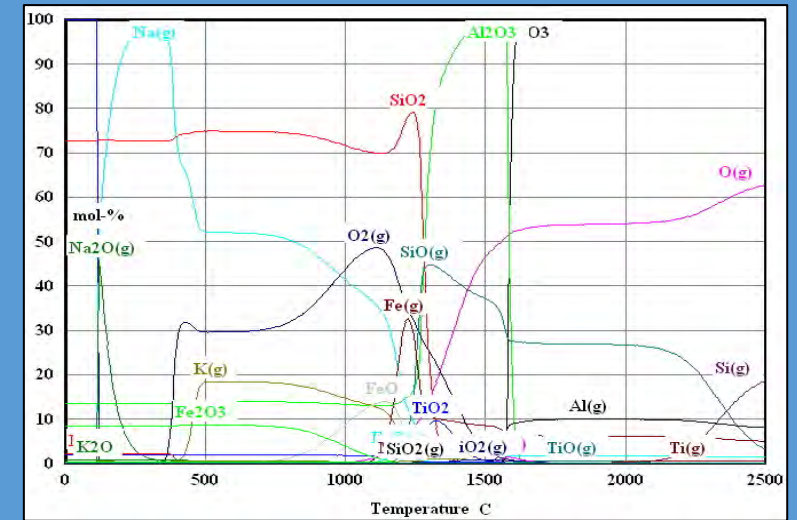
### Vacuum Fractional Thermal Column (VFTC)

- High Vacuum (at least E-4 torr)
- High temperature (up to 1600 C)
- Large regolith samples (up to 1 kilo)



0.18 torr, 1100 °C SP

0.25 torr, 1000 °C SP



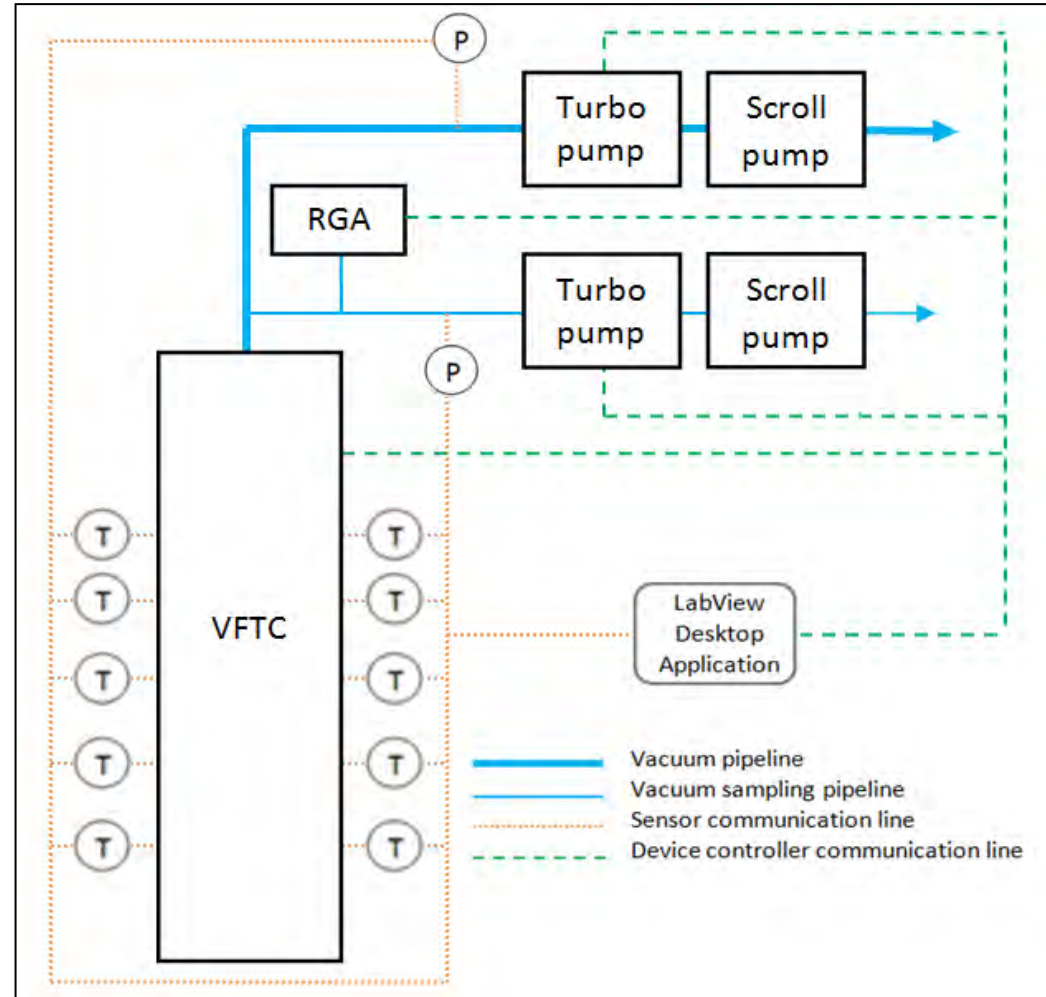
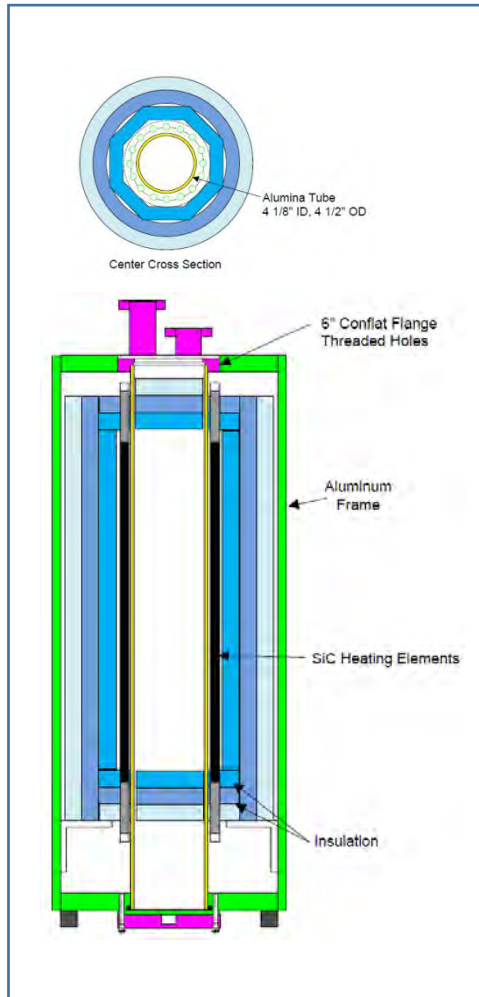


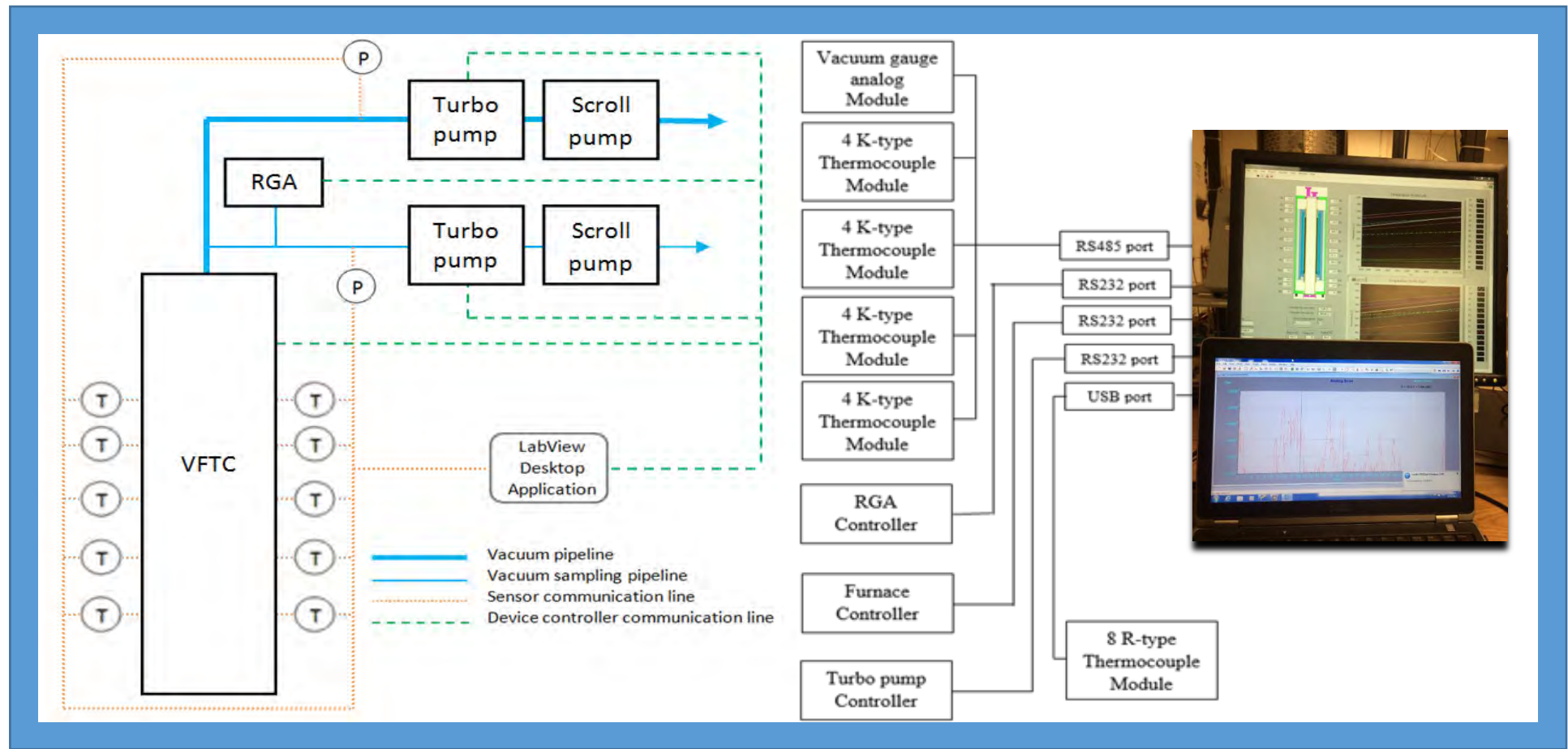
Research work currently being conducted at the Applied Research Laboratory (ARL) operated by Florida Institute of Technology (FIT) in Melbourne, Florida (USA).



**Vacuum Fractional Thermal Column (VFTC)** unit originally designed and built at NASA Kennedy Space Center (KSC) was substantially upgraded to perform the experimental research work.









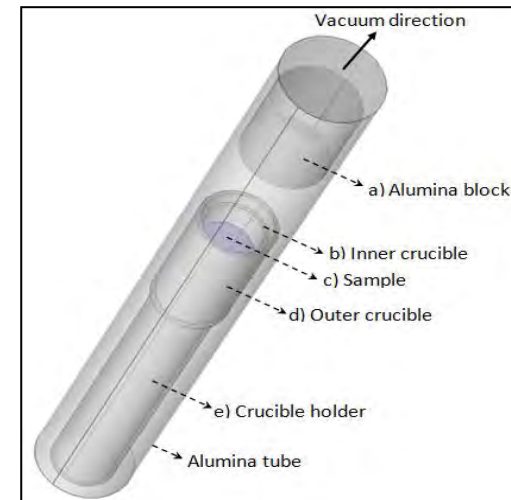
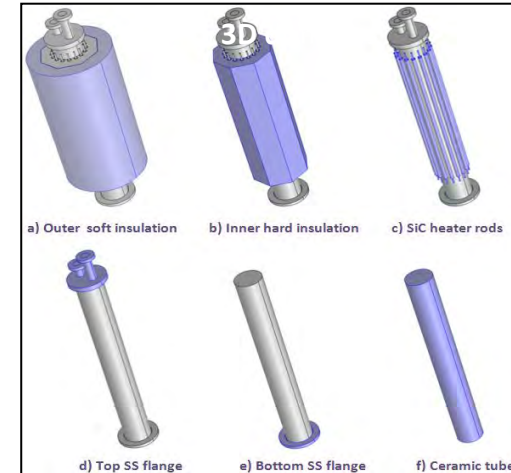


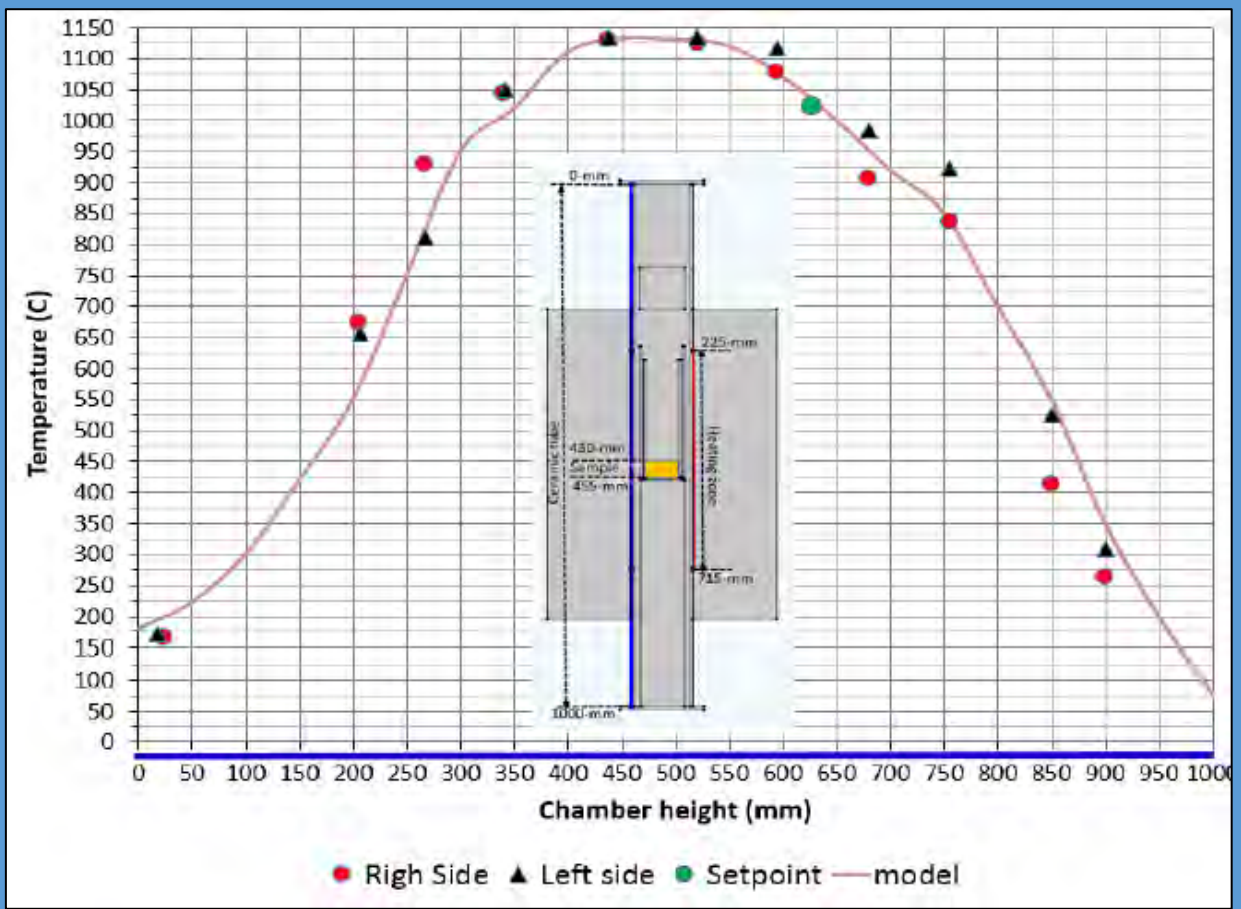
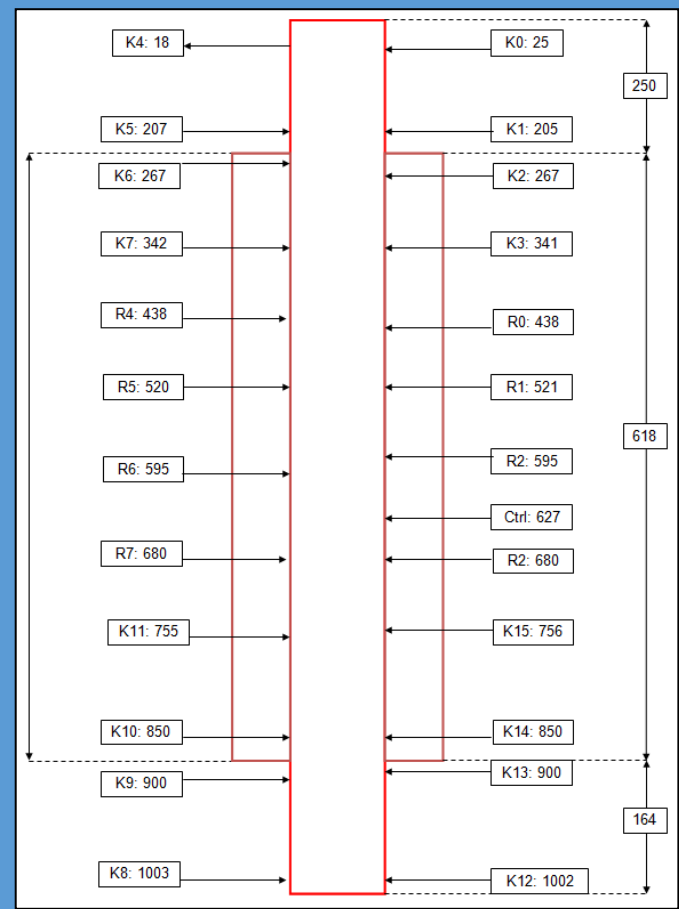
Measuring the temperature profile along the outer VFTC's wall at different heating target temperatures allows:

- **Validation** of a comprehensive 3D heat-transfer model.
- **Estimation** of the temperature profile throughout the sample inside VFTC

- COMSOL Multiphysics® is a general-purpose software platform, based on advanced numerical methods, for modeling and simulating physics-based problems.
- The mathematical models in COMSOL are discretized by the Finite Element Method (FEM), resulting in corresponding numerical models. The discretized equations are solved and the results are analyzed, hence the term finite element analysis.
- The description of the laws of heat transfer physics for space and time dependent problems are expressed in terms of *partial differential equations* (PDEs).

- 3D domains
- Regolith sample, the only thermal radiation participating medium
- Asymmetric temperature distribution throughout symmetric domains
- Heat flux generated by 16 heating rods simplified to a single heat flux applied to the outer surface of the tube.
- Isotropic thermal properties.
- Constant heat transfer coefficients.
- Negligible scattering coefficient, only a constant absorption coefficient used in thermal radiation in participating media





## JSC-1\*



Oxide	JSC-1 (mean of 3)		Lunar Soil 14163*	
	Conc.	Std. Dev.	Conc.	
	Wt %	Wt %	Wt %	
SiO <sub>2</sub>	47.71	0.10	47.3	
TiO <sub>2</sub>	1.59	0.01	1.6	
Al <sub>2</sub> O <sub>3</sub>	15.02	0.04	17.8	
Fe <sub>2</sub> O <sub>3</sub>	3.44	0.03	0.0	
FeO	7.35	0.05	10.5	
MgO	9.01	0.09	9.6	
CaO	10.42	0.03	11.4	
Na <sub>2</sub> O	2.70	0.03	0.7	
K <sub>2</sub> O	0.82	0.02	0.6	
MnO	0.18	0.00	0.1	
Cr <sub>2</sub> O <sub>3</sub>	0.04	0.00	0.2	
P <sub>2</sub> O <sub>5</sub>	0.66	0.01	---	
LOI	0.71	0.05	---	
<b>Total</b>	<b>99.65</b>		<b>99.8</b>	

LOI = Loss on ignition \* Papike et al (1982)

## JSC MARS-1\*\*



Oxide	VL-1	VL-2	Pathfinder	JSC Mars-1	
	Wt%*	Wt%*	Wt%**	Wt%***	Wt%****
SiO <sub>2</sub>	43	43	44.0	34.5	43.5
Al <sub>2</sub> O <sub>3</sub>	7.3	7	7.5	18.5	23.3
TiO <sub>2</sub>	0.66	0.56	1.1	3.0	3.8
Fe <sub>2</sub> O <sub>3</sub>	18.5	17.8	16.5	12.4	15.6
MnO	n.a.	n.a.	n.a.	0.2	0.3
CaO	5.9	5.7	5.6	4.9	6.2
MgO	6	6	7.0	2.7	3.4
K <sub>2</sub> O	<0.15	<0.15	0.3	0.5	0.6
Na <sub>2</sub> O	n.a.	n.a.	2.1	1.9	2.4
P <sub>2</sub> O <sub>5</sub>	n.a.	n.a.	n.a.	0.7	0.9
SO <sub>3</sub>	6.6	8.1	4.9	n.a.	n.a.
Cl	0.7	0.5	0.5	n.a.	n.a.
LOI	n.a.	n.a.	n.a.	21.8	n.a.
<b>Total</b>	<b>89</b>	<b>89</b>	<b>89.5</b>	<b>101.1</b>	<b>100.0</b>

\* Swamp Works lab at the NASA Kennedy Space Center

\*\* Electrostatics and Surface Physics Laboratory (ESPL) at the NASA Kennedy Space Center



Run #	Sample Mass (g)	Temp. SP (°C)	Max. Temp (°C)	Vacuum/Pressure (Torr)	Soak Time at SP (min)	Post Soak Vacuum Time (min)
1	510.0	700		0.06	120	0
2	500.1	900		0.06	120	0
3	499.3	1000	1097	0.54	120	15
4	500.3	1000	1155	0.25	180	0
5	500.1	1000	1157	0.22	180	120
6	500.1	1000	1174	760	180	0
7	500.1	900	1047	0.22	180	120
8	500.3	950	1091	0.2	180	120
9	125.2	1000	1105	0.2	180	120
10	125.0	1100	1152	0.2	180	120
11	125.1	1100		0.19	60	120
12	125.1	1025		0.18	180	120
13	125.8	1050	1112	0.18	180	120
14	125.2	1035		0.18	180	120
15	125.4	1075		0.19	180	120
16	125.4	1100		0.18	0	120



Tests conducted without RGA in operation

Interfacial phenomena (Surfatial Tension) occur in many high temperature processes such as steelmaking, welding etc.



a) Top view of crucible containing 500.1 grams of JSC-1 sample and filling 102 mm (4") of a 254-mm-high (10") crucible.

b) Top view of crucible after thermal treatment under run 5 conditions (1000 °C SP, 1116 max. temperature, and 0.22 torr vacuum). Labels: Melted material diffused from core section, Sintered top section.

c) Front view of the interior of JSC-1 sample after thermal treatment under run 5. Labels: 152 mm (Diffusion height), 102 mm (Original sample height).

d) Magnification of area marked with a red rectangle in c).


**JSC-1 sample obtained under run 5 conditions**



a) b) Top view of crucible after thermal treatment under run 5 conditions (1000 °C SP, 1116 max. temperature, and 0.22 torr vacuum).

c) d) Top view of crucible after thermal treatment under run 13 conditions.

**JSC-1 sample obtained under run 13 conditions.**



a) b) Top view of crucible after thermal treatment under run 10 conditions.

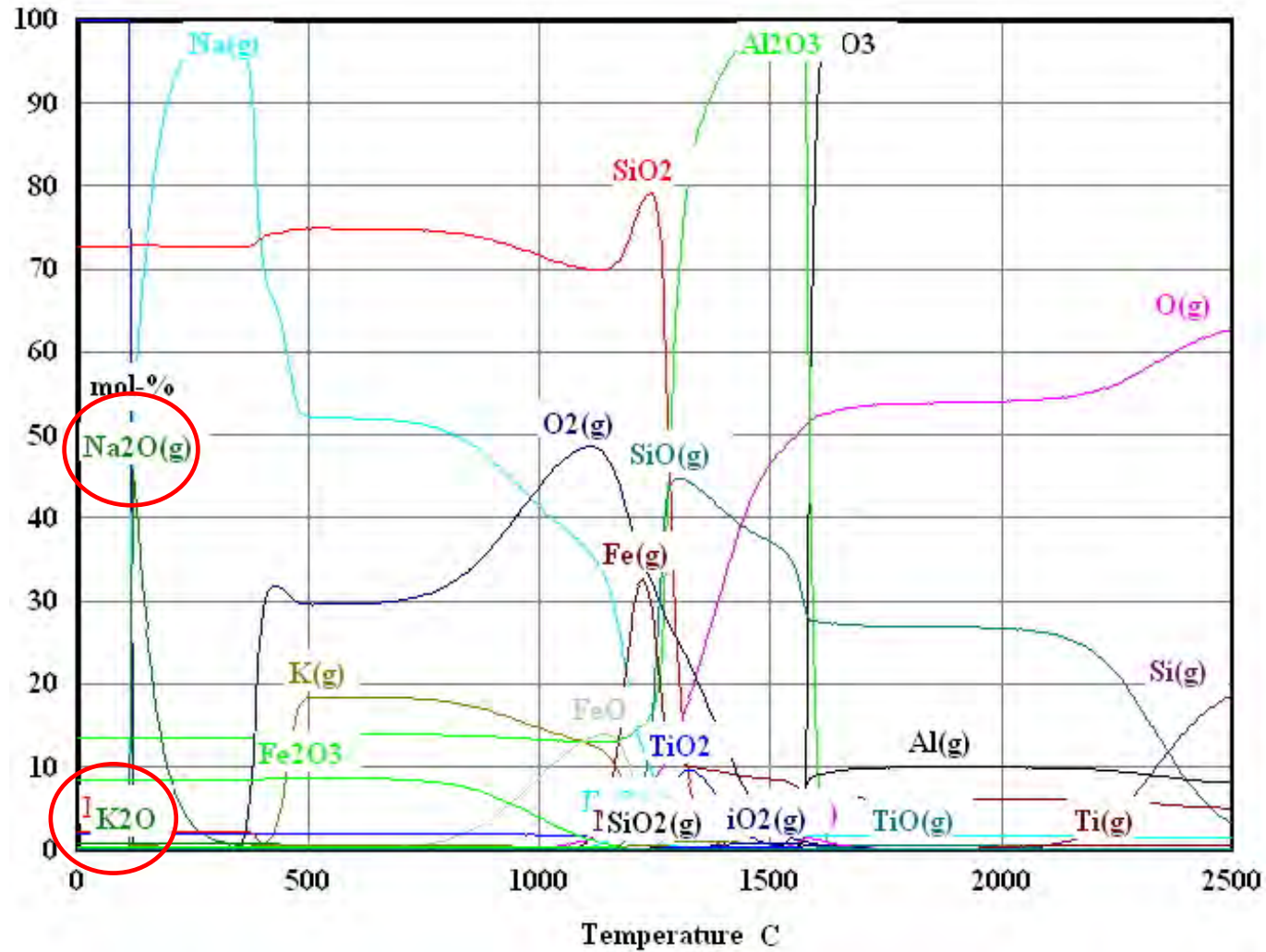
**JSC-1 sample obtained under run 10 conditions**

What is the driving force causing observed JSC-1 molten-phase diffusion/displacement?

- Pressure gradient along the crucible?
- Release of water vapor under vacuum?
- Metal oxide decomposition?
- Thermo diffusion due to temperature gradient at the surface and along the crucible?







$N_2O$  and  $K_2O$  the only two JSC-1 compounds to decompose at moderate vacuum



High temperature Interfacial phenomena (thermo diffusion) occur in many processes at high temperature..

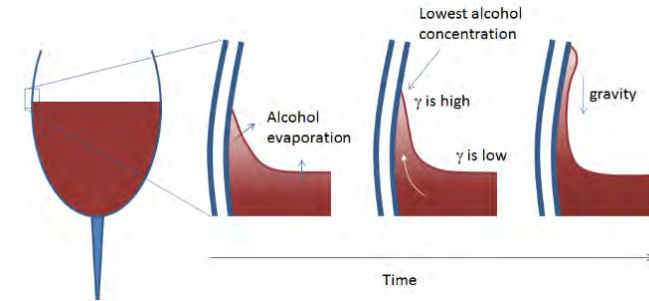
**Surface tension gradients can arise from:**

- (i) temperature differences along the surface which cause *thermo-capillary* flow;
- (ii) composition differences along the surface which cause *diffuso-capillary* flow;
- (iii) electrical potential differences along the surface which cause *electro-capillary* flow.

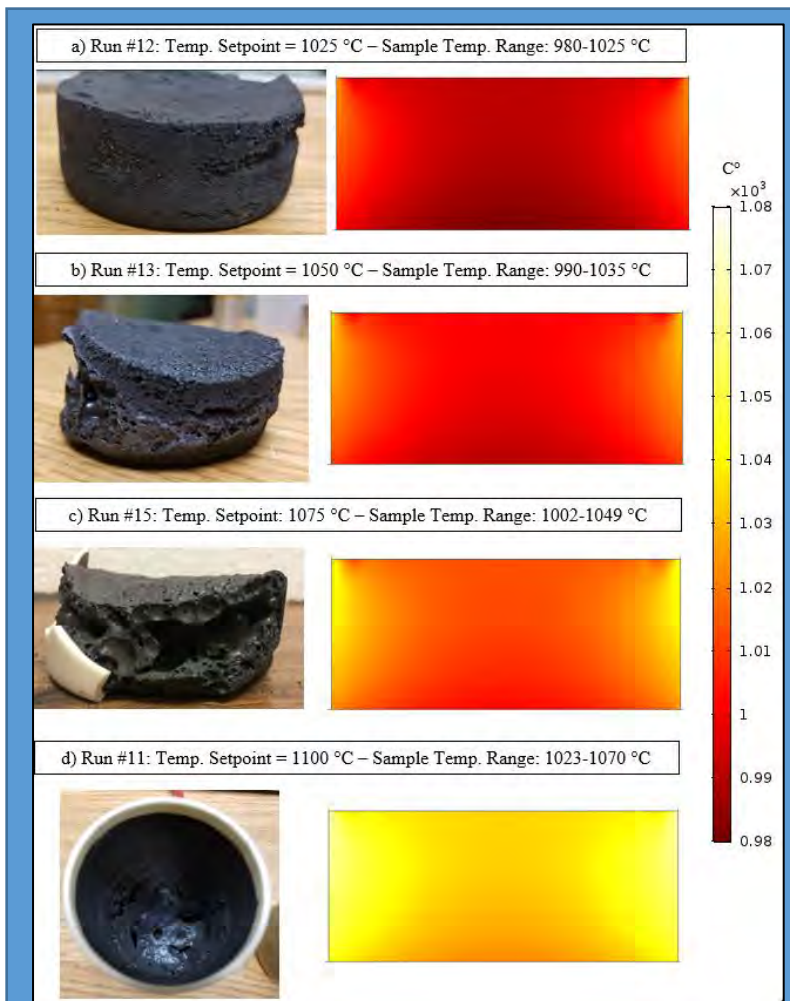


JSC-1 sample obtained under run 10 conditions

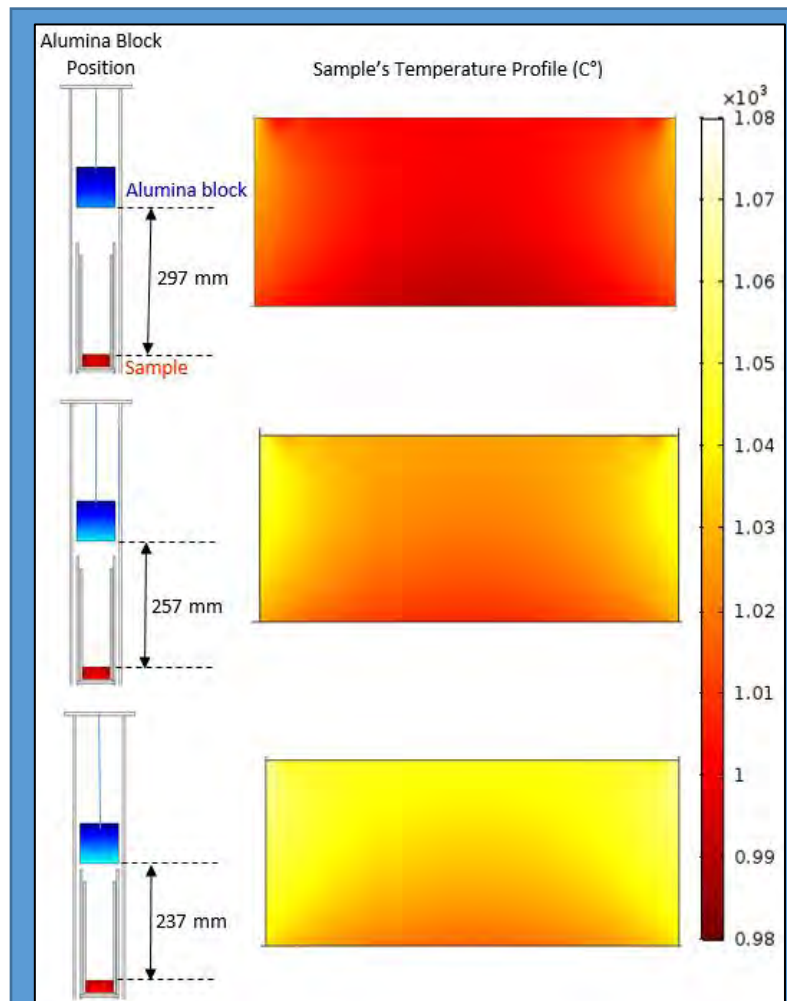
*Tears of wine form due to the surface tension gradient between the meniscus and the flat surface of the wine (Marangoni effect)*







Estimated temperature distribution within the sample as SP temperature increases from 1025 to 1100 C under 0.2 torr vacuum.



Estimated temperature profile within the JSC-1 sample as the separation of the alumina block hanging from the top SS flange goes from 297 to 237 mm at 1025 °C under 0.2 torr vacuum.

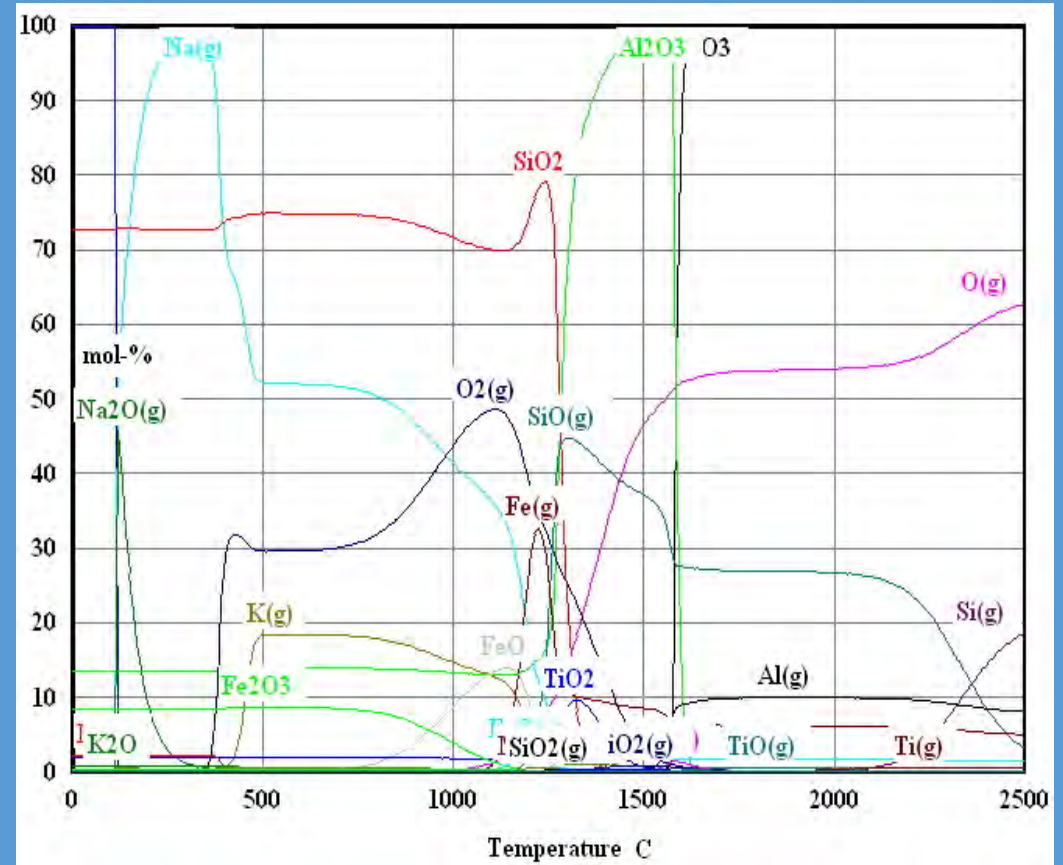
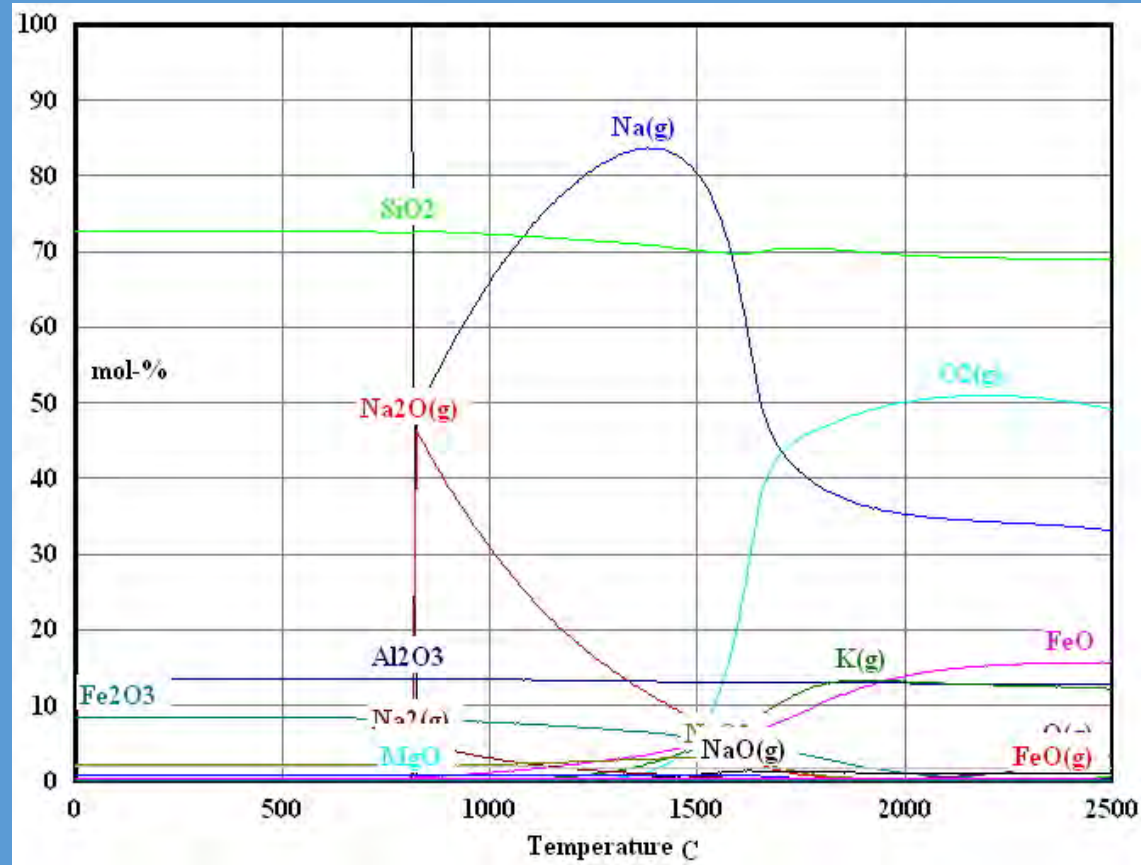
Run	Diameter (mm)			Average Diameter (mm)	Thickness (mm)			Average Thickness (mm)
	1	2	3		1	2	3	
KSC LD	73.5			73.5	25.5			25.5
KSC SD	62.0			62.0	27.0			27.0
KSC	59.3	63.7		61.5	25.7	25.7		25.7
3	57.9	61.0		59.4	25.0	24.5		24.8
6	60.0	57.0		58.5	24.5			24.5
8	60.5	62.0		61.3	26.5			26.5
11	60.0	60.5		60.3	23.5	23.3		23.4
12	69.8	61.5	61.0	64.1	27.0	24.5	32.5	28.0
13	65.0	65.8		65.4	20.8	21.5	27.0	23.1

Run	Load (lbf)	Load (N)	Tensile Stress (psi)	Compressive Stress (psi)	Tensile Stress (kPa)	Compressive Stress (kPa)	Comments
KSC LD	657.0	2922.5	144.0	1439.7	992.7	9926.7	
KSC SD	3074.0	13673.8	754.2	7542.2	5200.1	52001.4	
KSC	1819.0	8091.3	472.7	4727.4	3259.4	32594.2	
3	N/A	N/A	N/A	N/A	N/A	N/A	Failed Preloading
6	1597.0	7103.8	457.6	4576.5	3155.4	31553.6	
8	N/A	N/A	N/A	N/A	N/A	N/A	Failed Preloading
11	190.0	845.2	55.4	554.1	382.0	3820.4	
12	1135.0	5048.7	259.8	2598.0	1791.3	17912.6	
13	N/A	N/A	N/A	N/A	N/A	N/A	Failed Preloading

Built at KSC under He



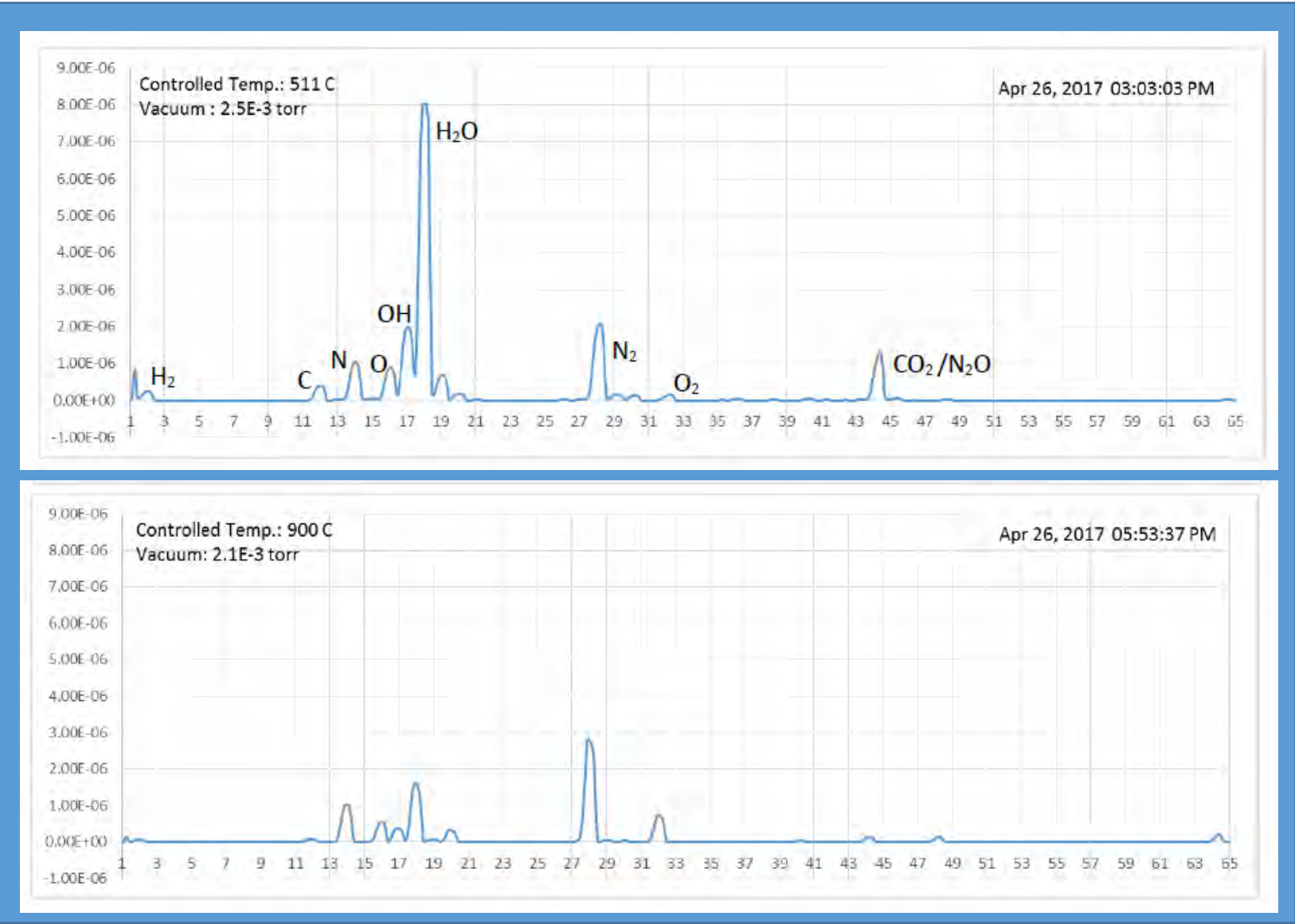
Mechanical tests performed on the sintered JSC-1 products yielded in runs 3, 6, 8, 11, 12, and 13.



Estimated\* decomposition of Simulant JS-1 at ambient pressure and high vacuum (10<sup>-7</sup> torr) respectively.

\*Estimation assuming thermodynamic equilibrium calculation available in HSC Chemistry® 6.0

# Residual Gas Analyzer (RGA) reading





- Acquire RGA's baseline
- Run a set of experiments using new higher vacuum level
- Acquire outlet gas composition via RGA to estimate JSC-1 decomposition (mainly O<sub>2</sub>)
- Evaluate results



- Improve high vacuum level
- Run experiments with JSC MARS-1 samples
- Include fractional separation capability inside VFTC to demonstrate the feasibility of using a terrestrial-precursor-free thermal-driven-based process to transform JSC-1 into O<sub>2</sub>, metals, and alloys.
- Evaluate and publish results

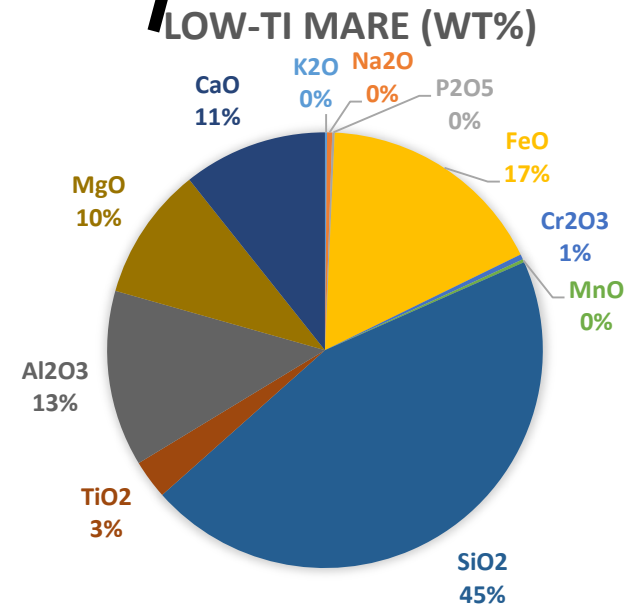
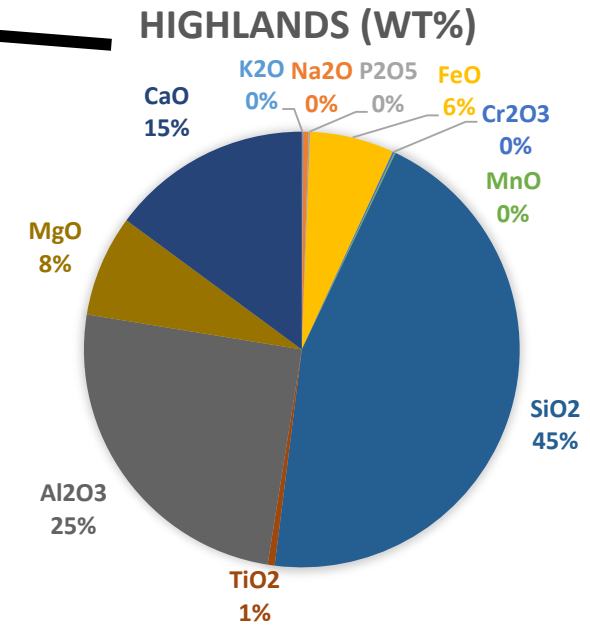
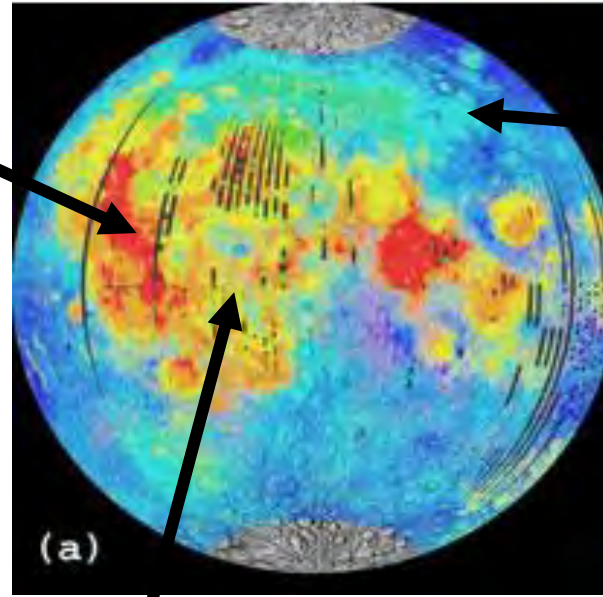
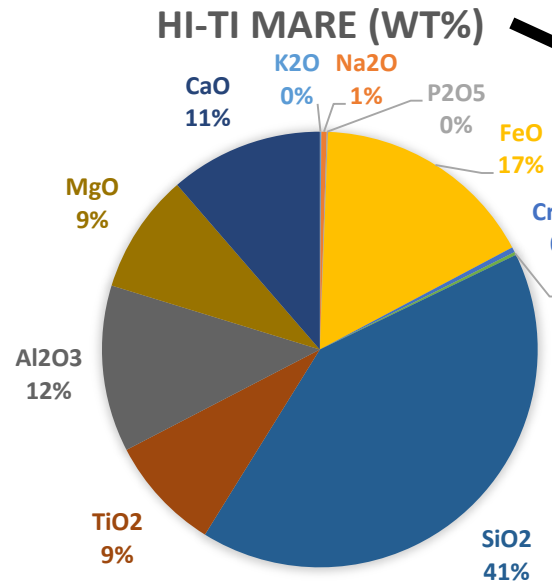
- Dr. Orlando Melendez (NASA KSC)
- NASA Florida Space Grant Consortium (FSGC)
- Mr. Fred Bristol (FIT Welding Shop) KSC
- Dr. James Mantovani (Swamp Works lab at NASA KSC)  
Dr. Carlos calle (ESPL at NASA KSC)  
Dr. Mark Nurge (Physics lab at NASA KSC)  
Dr. Richard Arkin (SGT at NASA KSC)

**THANKS!!!!**

**QUESTIONS?**

**BACKUP SLIDES**

# LUNAR REGOLITH COMPOSITION





# ISRU PROCESS CANDIDATES FOR O<sub>2</sub> EXTRACTION FROM LUNAR REGOLITH

**Table 3 Qualitative comparison of lunar oxygen processes**

Processes	Technology <sup>a</sup>	No. of steps <sup>b</sup>	Process conditions <sup>c</sup>	Feedstock <sup>d</sup>	Total	Rank
<b>Solid/gas interaction</b>						
Ilmenite reduction with H <sub>2</sub>	8	9	7	3	27	4
Ilmenite reduction with C/CO	7	8	7	3	25	7
Ilmenite reduction with CH <sub>4</sub>	7	8	7	3	25	8
Glass reduction with H <sub>2</sub>	7	9	7	6	29	2
Reduction with H <sub>2</sub> S	2	6	6	8	22	12
Extraction with F <sub>2</sub>	5	1	2	10	18	16
Carbochlorination	3	3	3	10	19	15
Cl <sub>2</sub> plasma reduction	4	5	5	10	24	9
<b>Silicate/oxide melt</b>						
Molten silicate electrolysis	6	8	5	10	29	3
Fluxed silicate electrolysis	6	6	5	10	27	5
Caustic dis. electrolysis	5	4	3	10	22	13
Carbothermal reduction	6	3	3	10	22	14
Magma partial oxidation	2	2	4	5	13	19
Li or Na reduction of ilmenite	2	3	5	2	12	20
<b>Pyrolysis</b>						
Vapor pyrolysis	6	8	6	10	30	1
Ion plasma pyrolysis	4	8	4	10	26	6
Plasma reduction ilmenite	7	8	6	3	24	10
<b>Aqueous solution</b>						
HF acid dissolution	5	1	2	10	18	17
H <sub>2</sub> SO <sub>4</sub> acid dissolution	5	3	3	5	16	18
<b>Coproduct recovery</b>						
H <sub>2</sub> -He-water production	7	9	7	1	24	11

<sup>a</sup>Technology readiness: 1 = major technologic development required; 10 = no major unknowns.

<sup>b</sup>No. of steps: 1 = many (>5); 10 = one step.

<sup>c</sup>Process conditions (temperature, energy, plant mass, corrosion): 1 = severe; 10 = low.

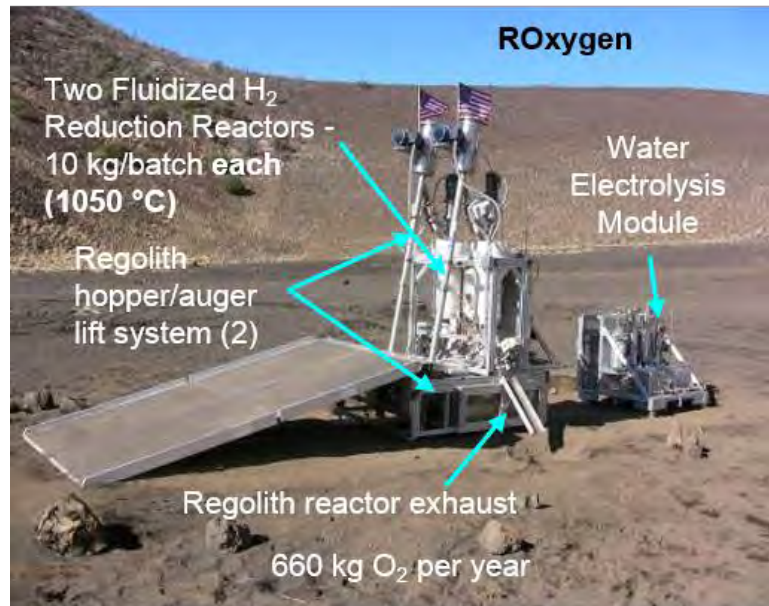
<sup>d</sup>Feedstock requirements: 1 = huge quantities; 2 = mare, beneficiated (ilmenite); 5 = mare unbeneficiated; 10 = any feedstock, unbeneficiated.

# ISRU PROCESS CANDIDATES FOR O<sub>2</sub> EXTRACTION FROM LUNAR REGOLITH:

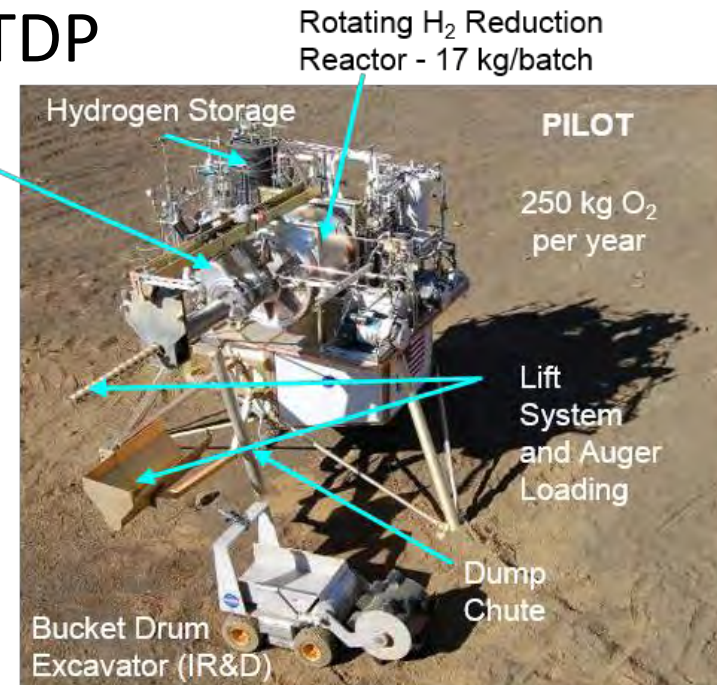
## H<sub>2</sub> REDUCTION



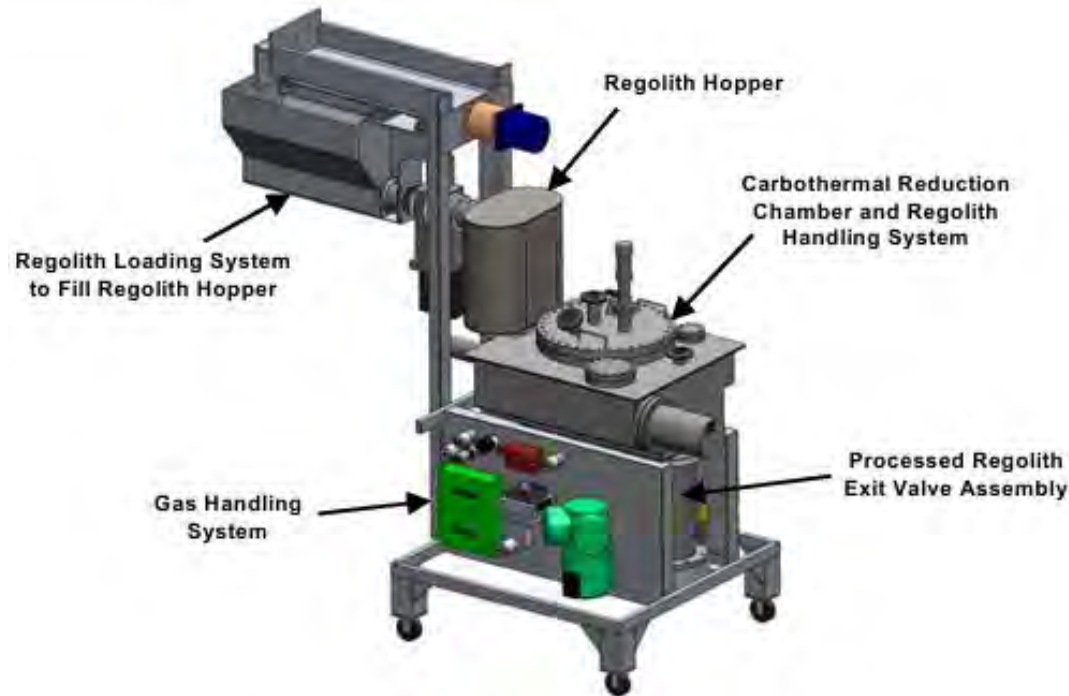
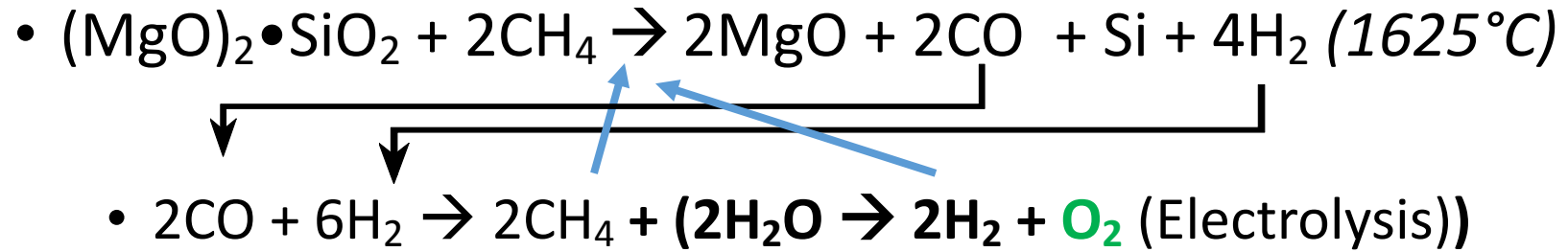
- Hardware demonstrations under ETDP



1. Heat Regolith to >900 C
2. React with Hydrogen to Make Water
3. Crack Water to Make O<sub>2</sub>



# ISRU PROCESS CANDIDATES FOR O<sub>2</sub> EXTRACTION FROM LUNAR REGOLITH: **CARBOTHERMAL REDUCTION**



**Figure 6a. Orbitec Carbothermal Reduction System**



**Figure 6b. PSI Solar Concentrator Breadboard**



# ISRU PROCESS CANDIDATES FOR O<sub>2</sub> EXTRACTION FROM LUNAR REGOLITH: **MOLTEN REGOLITH ELECTROLYSIS (MRE)**

- $M_xO_y \rightarrow xM + \frac{y}{2}O_2$  (Molten Electrolysis)
  - $2FeO \rightarrow 2Fe + O_2$ ;  $SiO_2 \rightarrow Si + O_2$
- Hardware maturation by KSC+MIT+Ohio

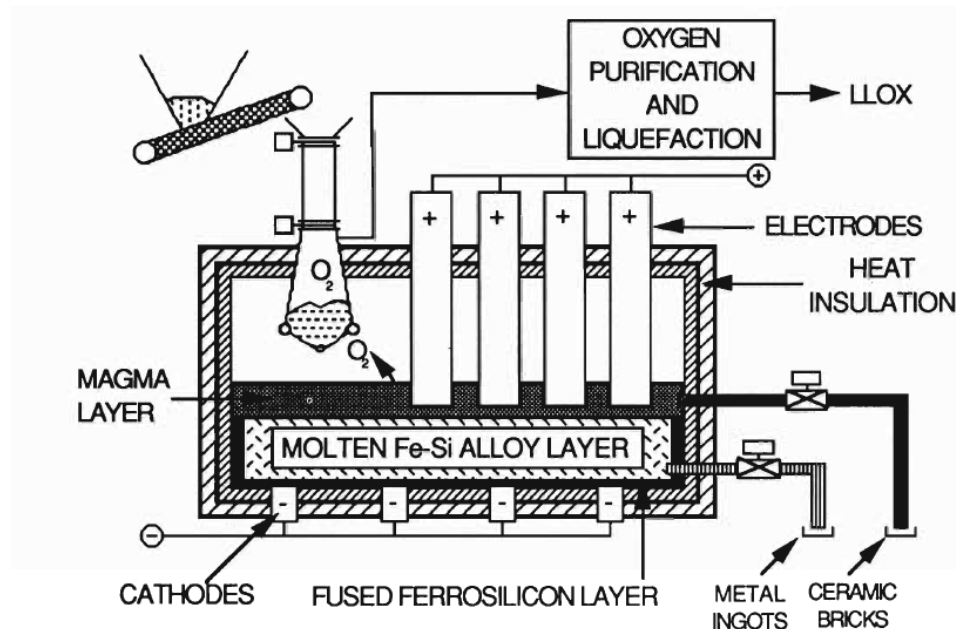
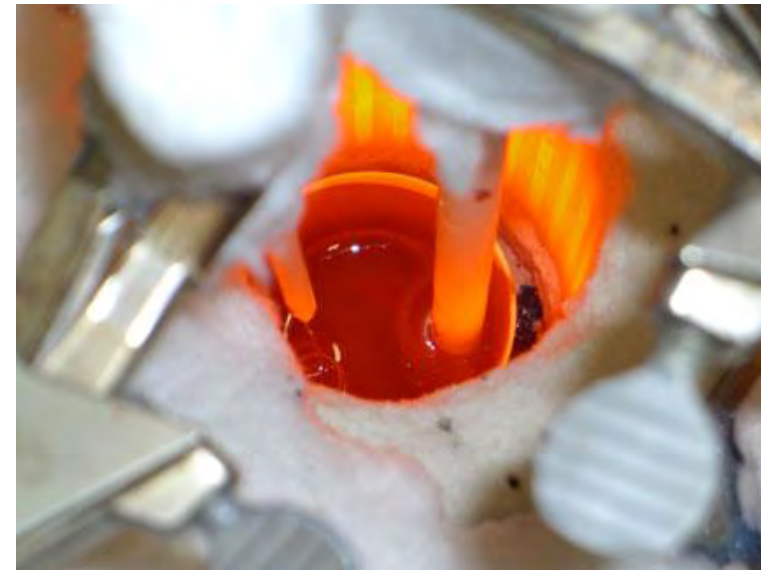


Figure 5. Magma electrolysis cell design for the production of LLOX from lunar regolith, modified after McCullough and Mariz (1990).



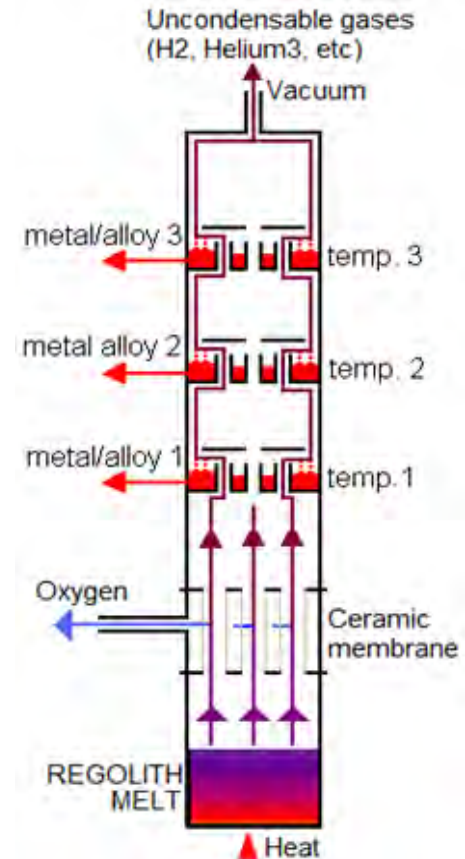


# NEW ALTERNATIVE ISRU PROCESS: **REGOLITH FRACTIONAL DESTILLATION (RFD)**

## Justification

Thermal decomposition of regolith and water/CO<sub>2</sub> ice under vacuum has important advantages when compared with H<sub>2</sub> reduction, carbonthermal reduction, and MRE:

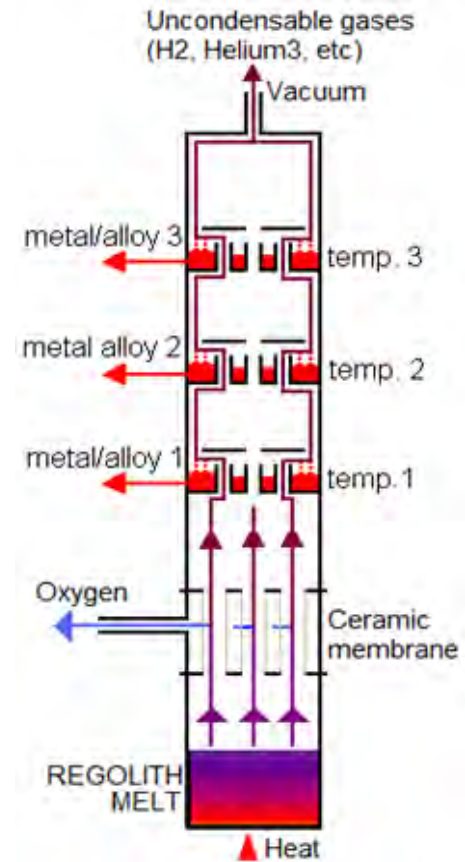
- it is a much simpler thermal-driven process.
- does not require terrestrial precursors.
- Uses an in-situ condition (high vacuum) as a driving force for the separation
- has the potential of separating O<sub>2</sub> and the metals simultaneously.
- Fractional separation allow selective production of different metals and alloys.
- can recover water and valuable incondensable gases, such as H<sub>2</sub> and Helium-3 if present in the regolith.



# NEW ALTERNATIVE ISRU PROCESS: RFD

## Justification (cont.)

- RFD does not need terrestrial precursors while H<sub>2</sub> reduction and carbonthermal processes do need terrestrial precursors (H<sub>2</sub> and CH<sub>4</sub> respectively).
- Both processes yield oxygen in water form requiring an electrolysis unit that also demands a water contaminant removal unit to the process.
- MRE does not need terrestrial precursors and generates oxygen but:
  - a) consumes inert electrodes that have to survive harsh conditions of the molten phase.
  - b) its yield is limited by selective molten solidification as the melt is depleted from the metal oxides.
  - c) Electrolysis of molten phase adds a larger complexity to the process (O<sub>2</sub> generation and Joule-heating).



# NEW ALTERNATIVE ISRU PROCESS: RFD

## Key Technological Challenges for Proof of Concept

- 1) Oxygen and hydrogen need to be removed from the gas phase immediately after the decomposition/gasification of the regolith and the water/CO<sub>2</sub> ice to avoid the regeneration of the metal oxides and water/CO<sub>2</sub> during condensation.
- 2) The condensation of the metals and alloys from the gas phase depleted in O<sub>2</sub> (and H<sub>2</sub> if needed) from (1) requires an understanding of gas-liquid equilibrium behavior of metal-based multi-component systems at vacuum conditions to separate the different metals and alloys based on their condensation points.

