National Aeronautics and Space Administration

Molten Regolith Electrolysis: Core Sample Analysis

Game Changing Development Program

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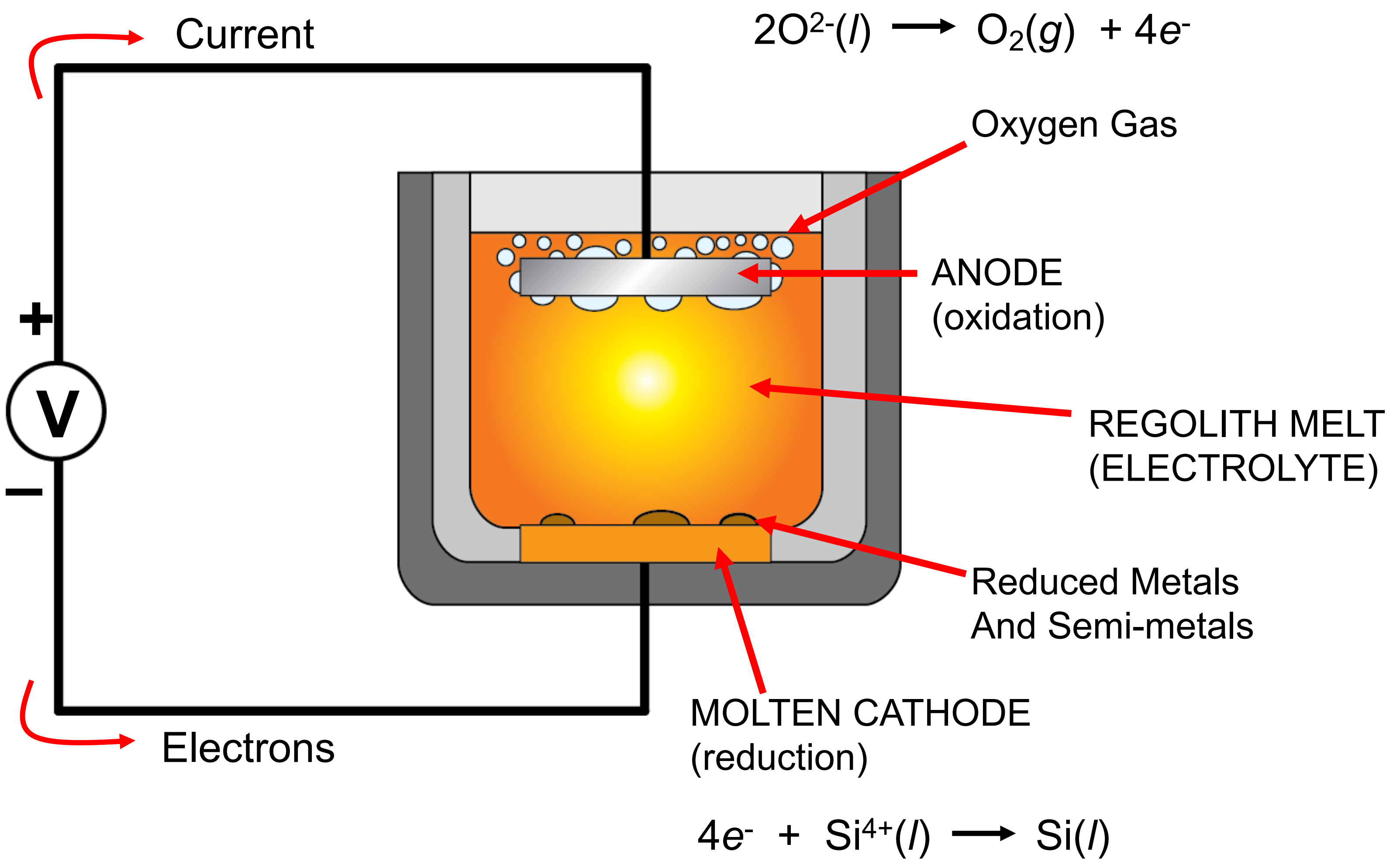
Molten Regolith Electrolysis Overview

<u>THREE CRITICAL PHASES</u>
1. Regolith heating to ≈1400˚C
2. Electrolysis allows the heater power to be reduced, and Joule-heating (resistive heating) sustains a melt temp of ≥1600˚C releasing molecular oxygen as bubbles from the melt at the anode.
3. Cool down solidifies the melt.

<u>NOTE:</u>
-Each oxygen molecule formed requires the removal of 4 electrons. *<u>This is a hard limitation</u>*.
-Amount of electrical current into the reactor corresponds to a theoretical limit to the maximum oxygen production rate.



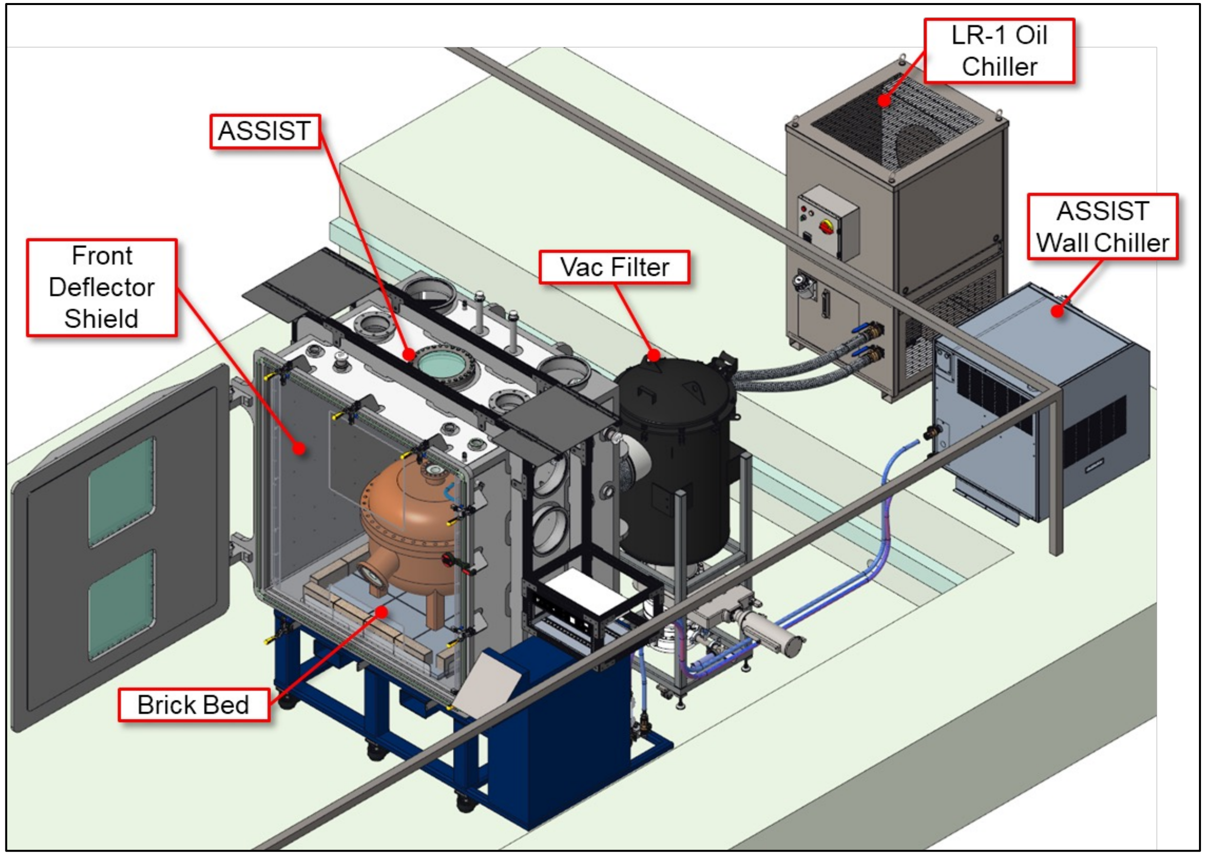
Test Information

- 25 kg of ICN-LHT-1G lunar regolith simulant was processed in the Lunar Resources, Inc. reactor under vacuum in the KSC Atmospherically Sealed Simulator for In-situ System Testing (ASSIST) chamber.
- Joule heating was sustained by electrolysis with temperatures ~1700°C and a maximum power draw of 9 kW during vacuum operations.
- Approximately 0.3 moles of O_2 per kW-hr measured, with an average rate of 0.067 kg/hr O_2 production.
- The extracted core sample was approximately 3 inches in diameter, 4 inches tall, and included anode, magma melt solid, and metal cathode material. The coring equipment used was a Bluerock 3-inch diamond concrete coring bit.

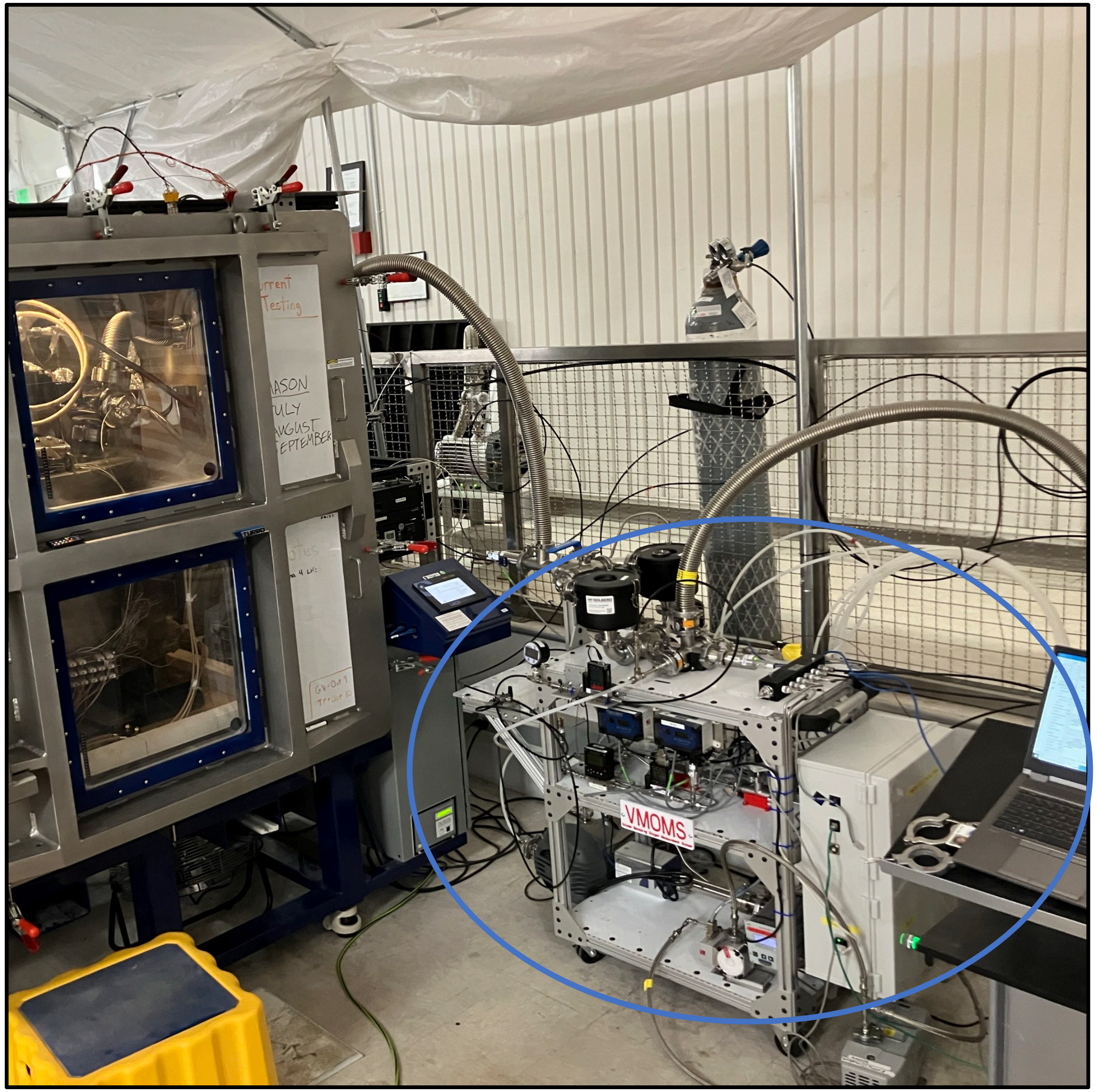
Weight %	**ICN-LHT-1G [Ref: APL SAR 2021]**
SiO_2	48.0
TiO_2	2.90
Al_2O_3	22.90
FeO	5.70
MnO	0.0
MgO	1.90
CaO	15.9
Na_2O	2.60
K_2O	0.0
P_2O_5	0.0

Composition of ICN-LHT-1G per Simulants Reference Guide. Si and Al are the most abundant oxides present.

MRE Test Set Up

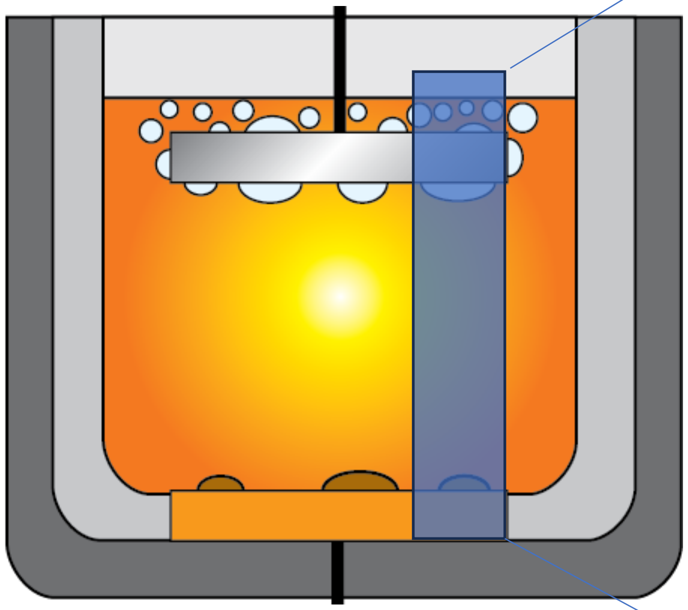


LR-1 Installation in KSC ASSIST Vacuum Chamber.



KSC VMOMS System

Core Removal and First Analysis: CT Scan



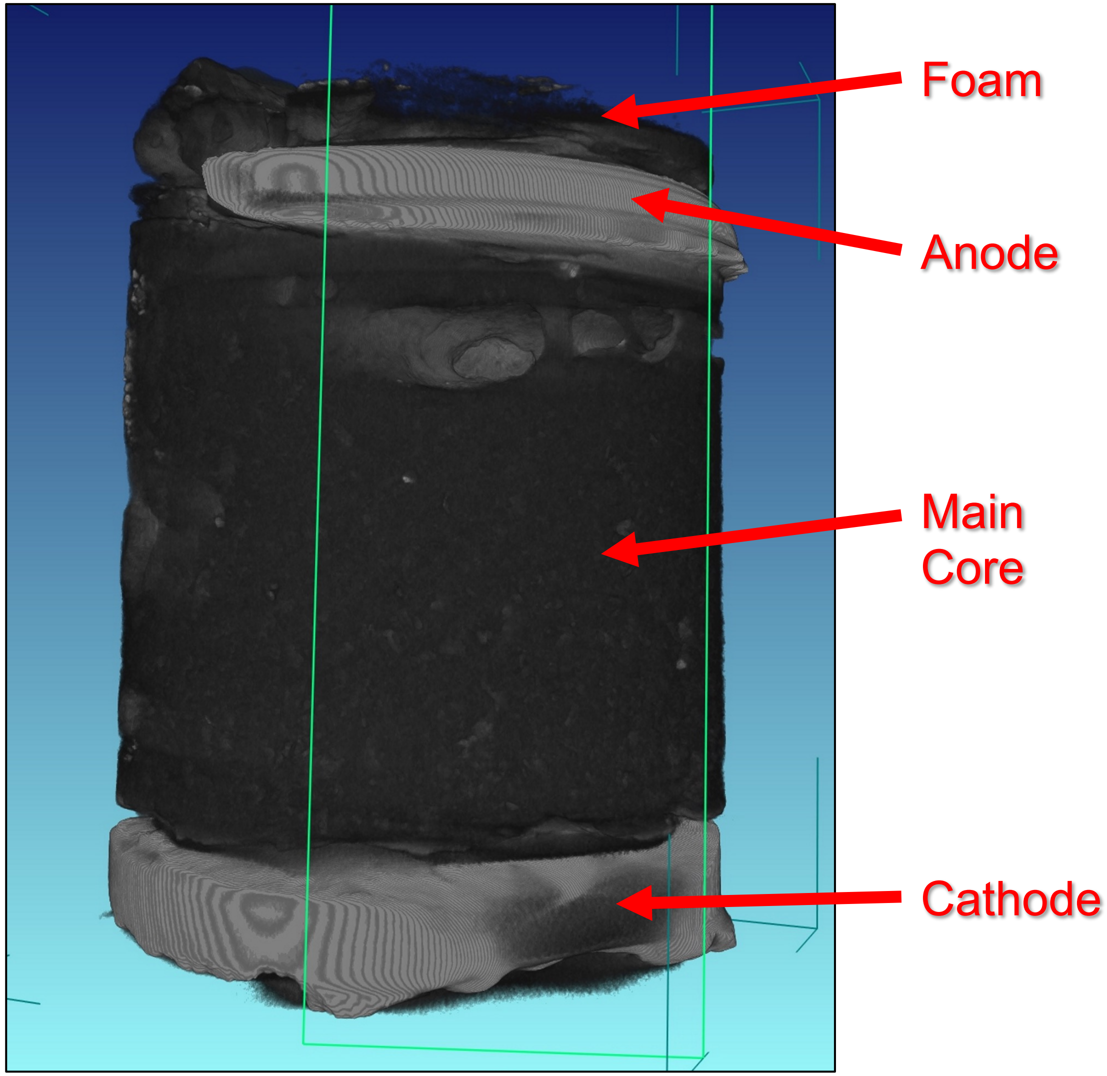
X-ray on the reactor was also performed. Results were limited; however, hardware that was identified appeared to be intact.

CT: North Star Imaging unit with its Microfocus Digital Radiography and Computed Tomography System (NSI X5000 DR/CT).

CT Scan of Complete Core

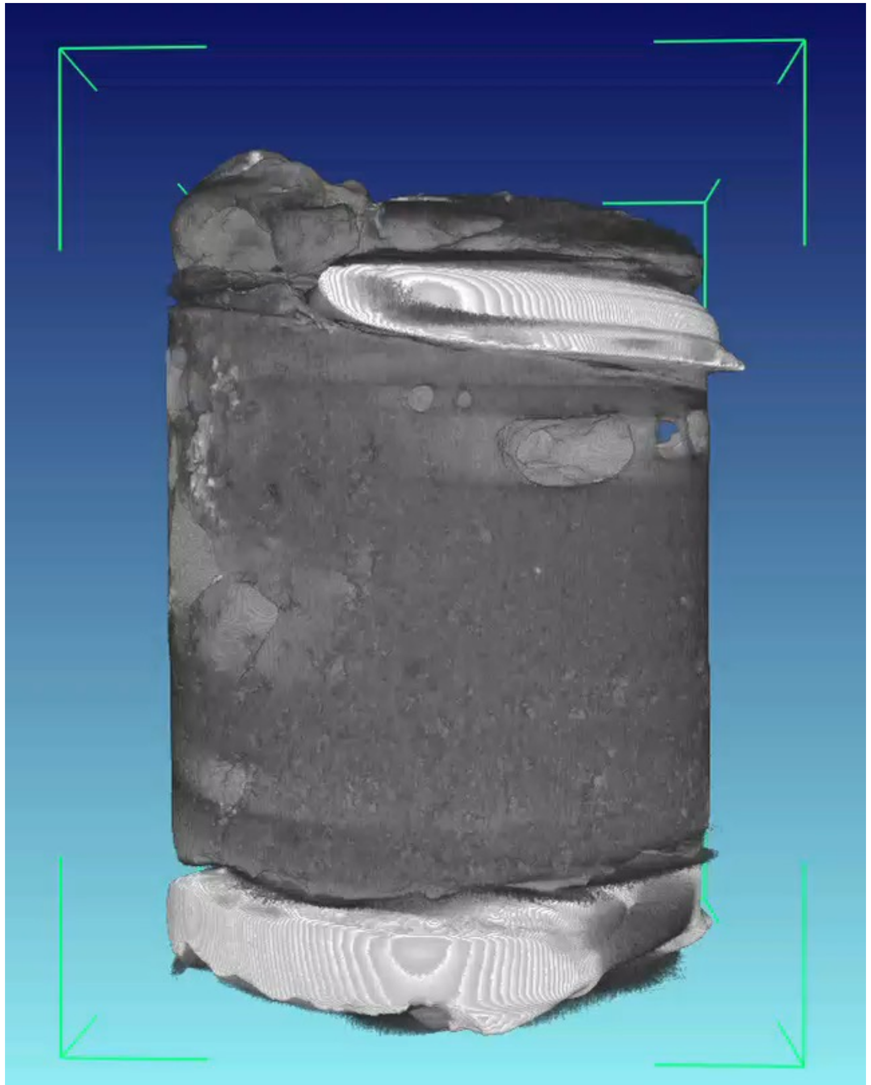
The entire core sample (all pieces) were stacked and analyzed together via CT imaging.

- Provided complete 3-dimensional data of the core.
- Allowed full cross-sectional data.



Core Surface CT Image

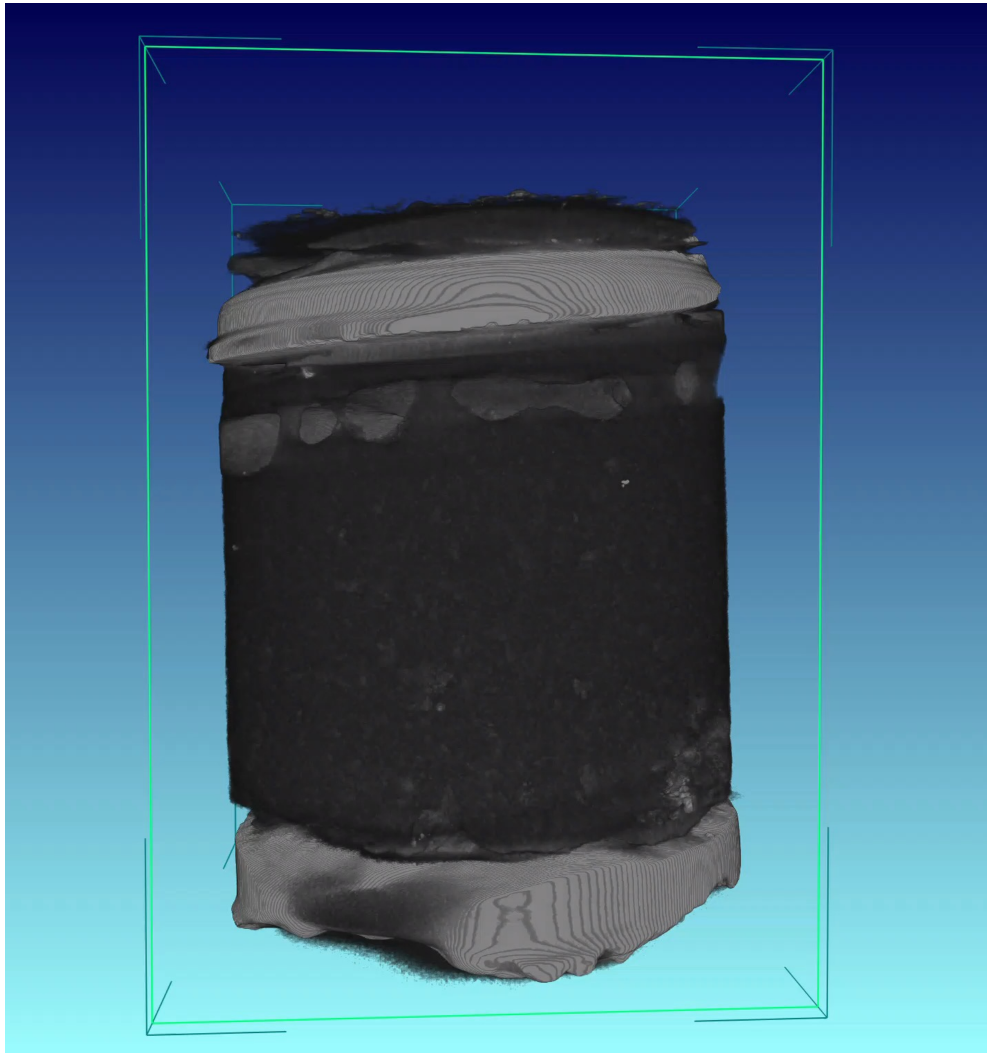
CORE CT Fly-by, and Core Slice Cut

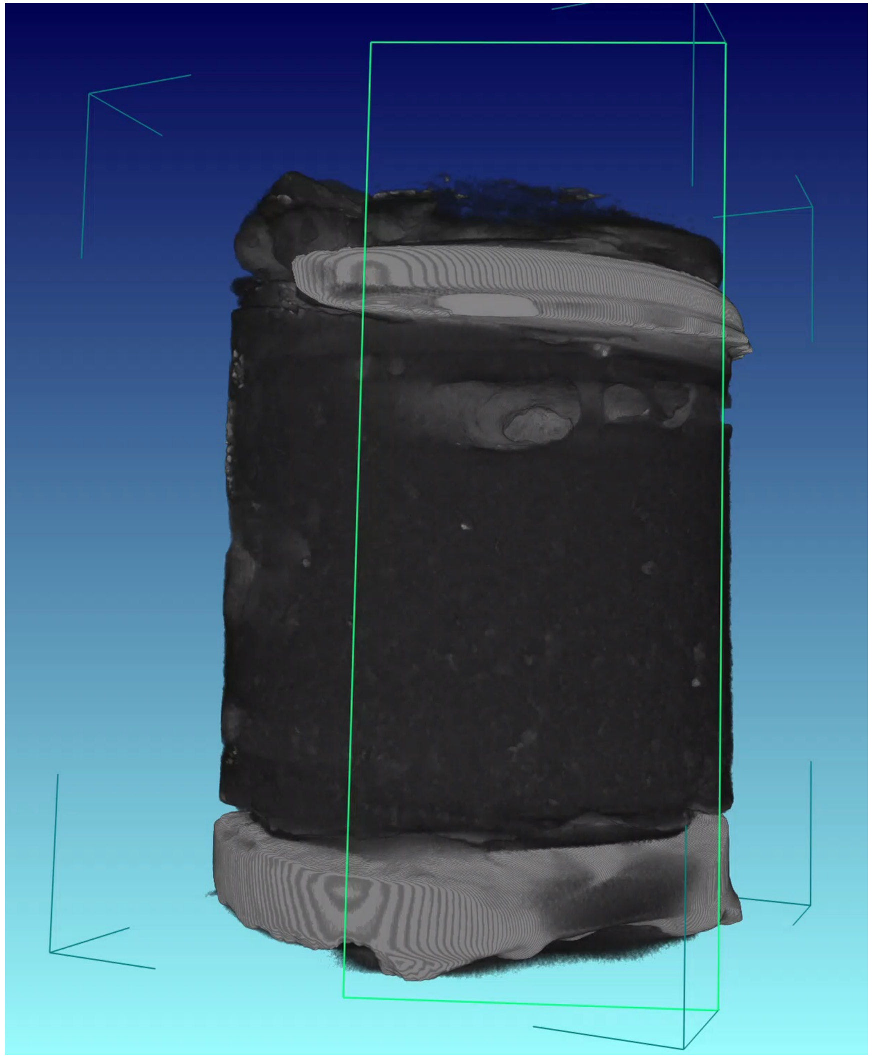


Core slice further used for EDS Mapping.

MRE Core Flythrough Videos, Cont'd

- Voids in solidified core, especially under anode.
- Substantial voids also present throughout core sample.

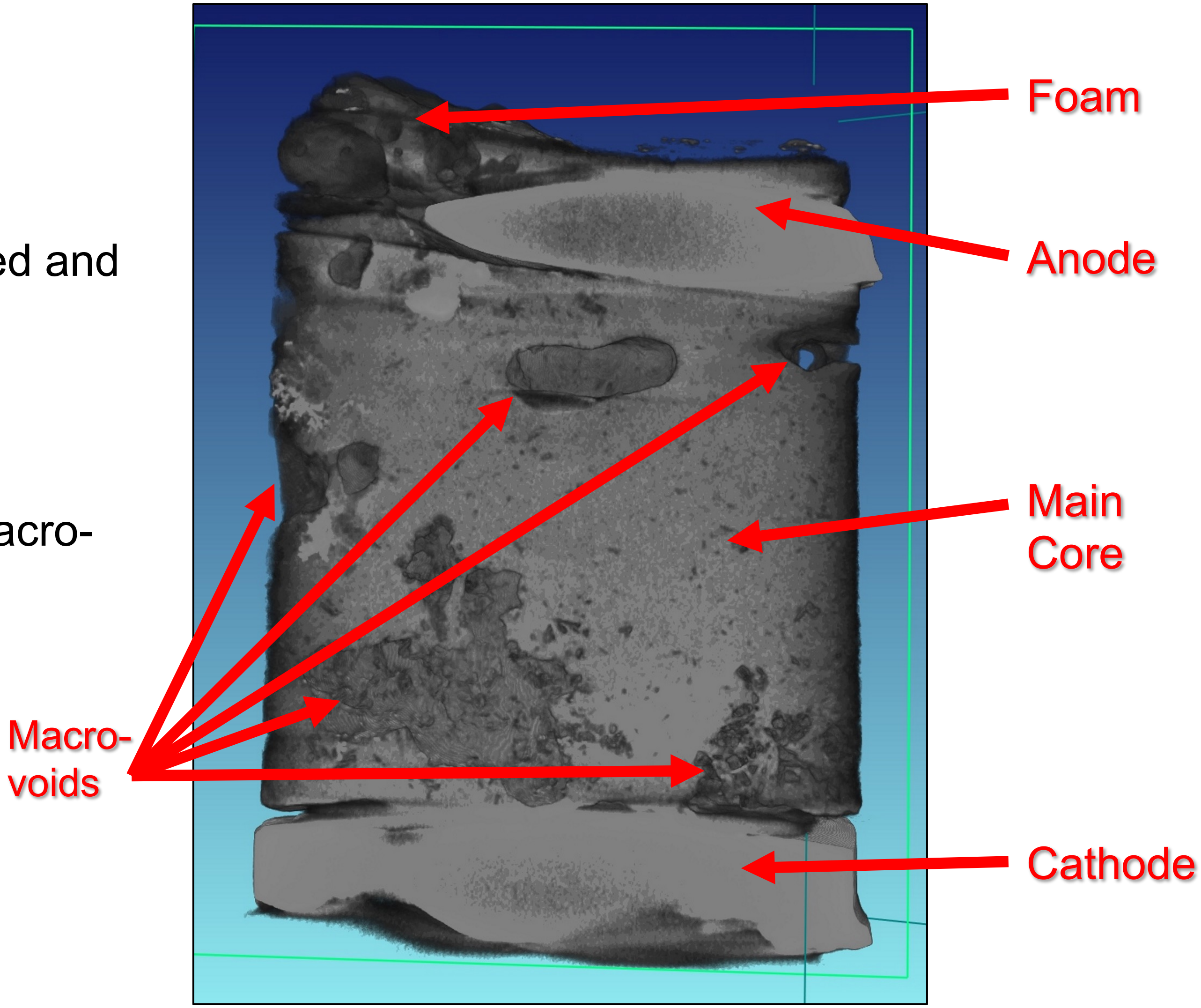




CT Scan of Core Main Piece

The entire core sample (all pieces) were stacked and analyzed together via CT imaging.

- Calculated the total volume of the main core piece.
- With mass, this allowed calculation of the macro-density of the main core piece.
- Calculated the macro-void volume.



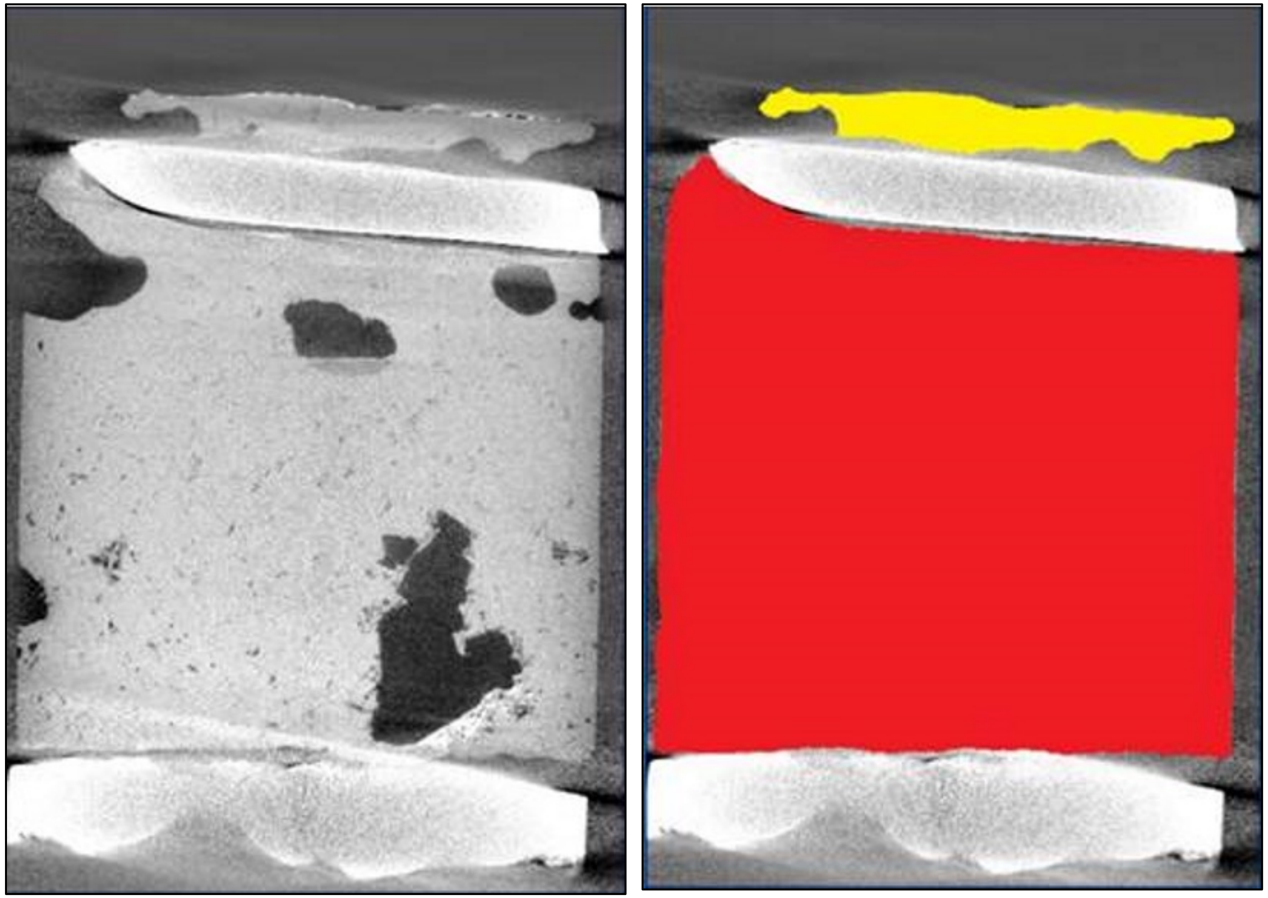
Core cross-section CT image showing macro-pores.

Total Core Volume Determination

- Utilized graphical analysis to determine total volumes of main core piece and top ‘foam’ piece.

Main Core Mass = 631.0 g
Foam Mass = 58.9 g
Main Core Total Volume = 9.714 cm^3
Foam Total Volume = 0.578 cm^3

Main Core Total Density = 2.35 g/cm^3
Foam Total Density (no voids) = 2.46 g/cm^3



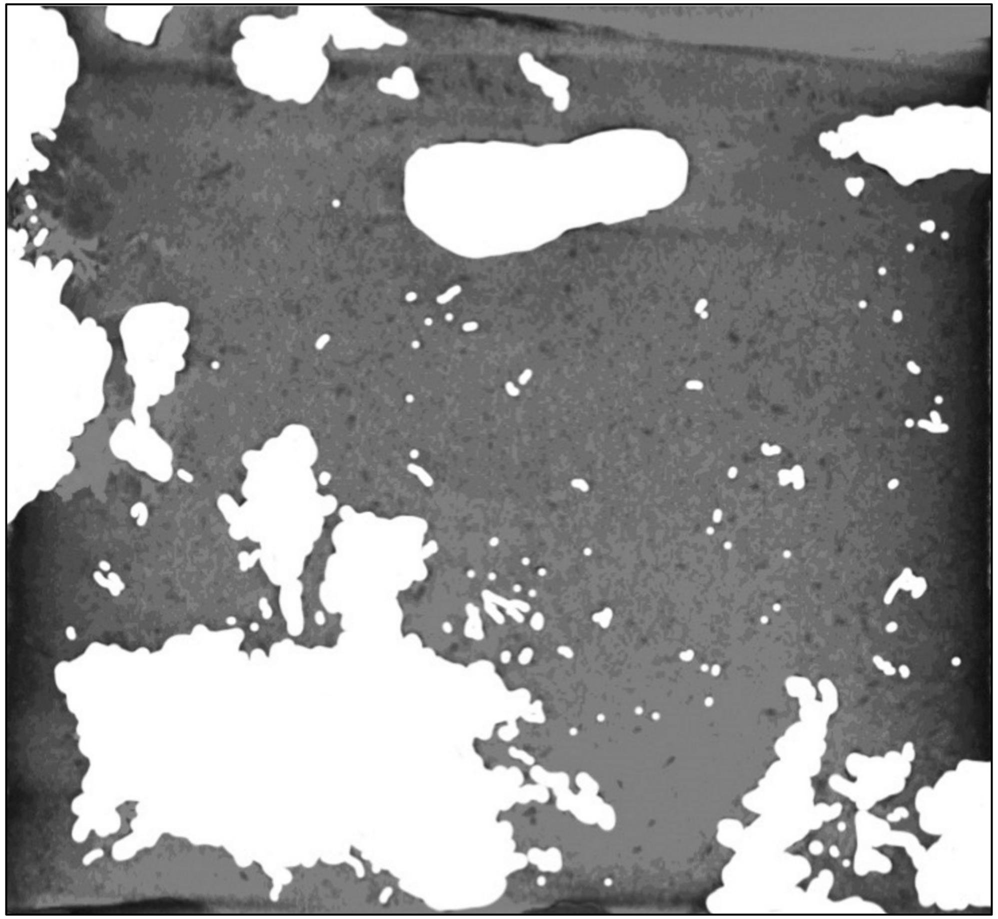
(L) Core cross-section CT image. (R) Mask applied to the Main Core (red) and Foam (yellow); masks were used to calculate the area of each cross-section increment.

Macro-Porosity Measurement

Porosity of the main core was measured via graphical analysis of the CT data.

Results

1. The porosities of each slice varied significantly (from 5.3% to 25.8%).
2. The overall porosity = 14.84%. Must be considered a minimum (did not include micropores as observed in SEM imaging).



Main Core cross-section CT image with white masks applied to macro-pore locations used to calculate the pore area of each cross-section increment.

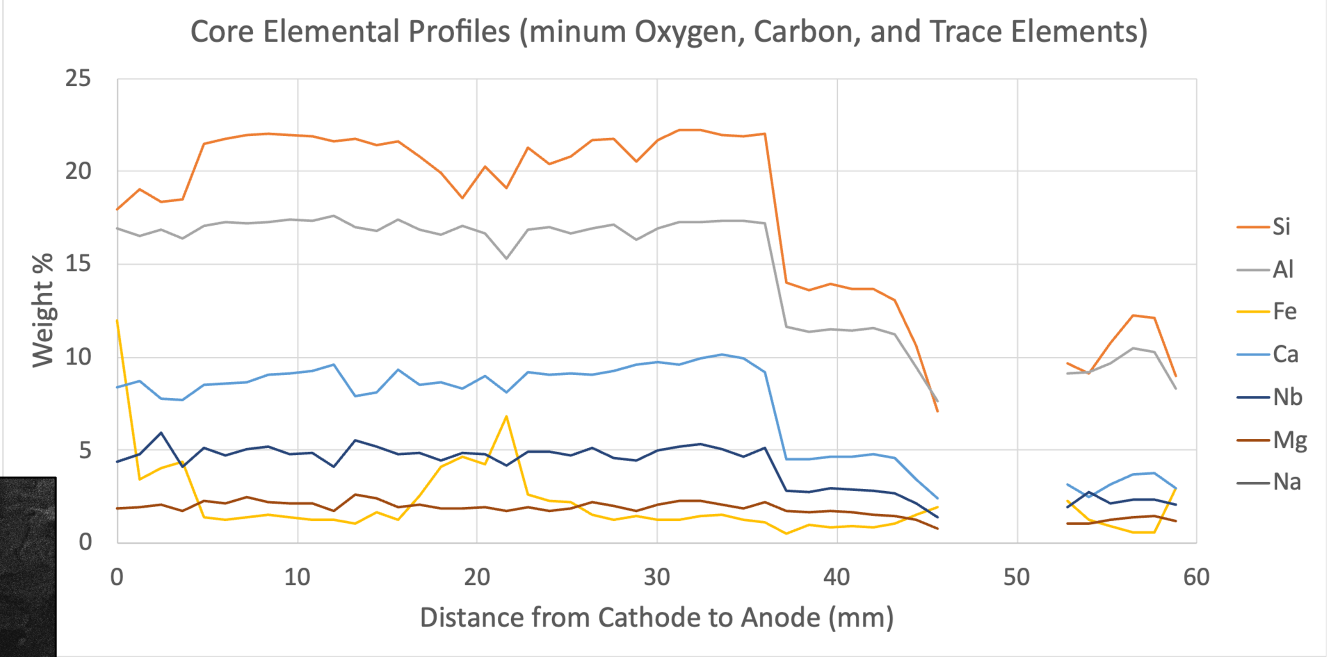
SEM/EDS of Core Main Piece

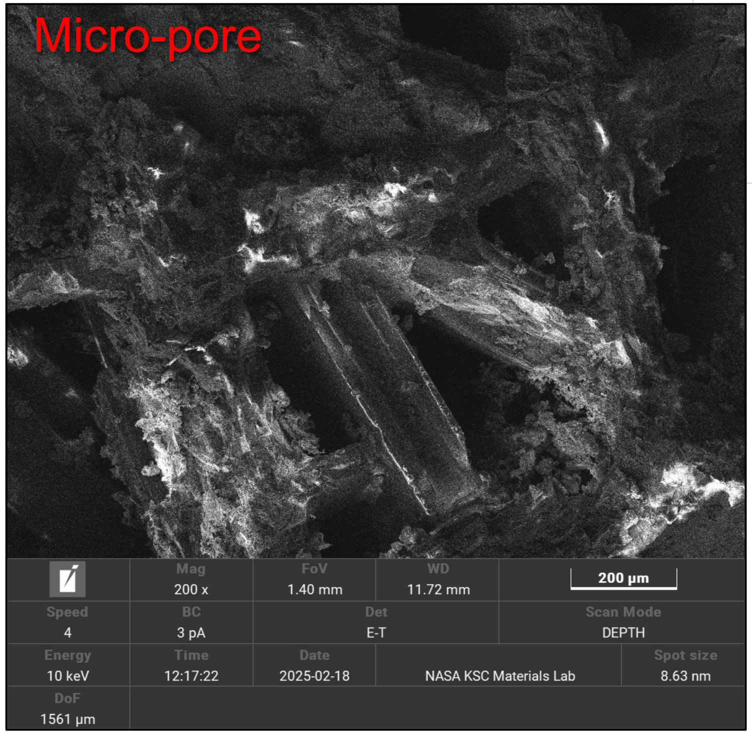
Scanning electron microscopy (SEM) of the whole main core was performed using a Tescan Model S8252G SEM with an Oxford Instruments Ultim Max 100 energy dispersive spectrometer (EDS) which allowed the entire main core piece to be analyzed.

Sampling was performed along a line that sampled the entire vertical depth of the core (as considered from its original geometry in the reactor) in 1.2 mm increments.

SEM/EDS of Core Main Piece

- SEM and EDS data were collected along the long axis at 1.2 mm increments.
- Micropores and local crystallites were observed.
- Increased iron content at the cathode end was observed.
- Niobium was detected across entire core.
- Increased C content toward anode end.





Average Elemental Composition (Oxford EDS)			
Element	Wt%	Element	Wt%
O	40.35	Nb	4.10
Si	18.02	Mg	1.83
Al	14.87	Mn	0.04
Fe	2.13	Si	0.02
Ca	7.33	Cl	0.01
Na	11.26	Zn	0.02
TOTAL	99.97		

KPP1: Mass of Remnant/Mass Loss

Total melt volume was estimated from CAD data for the reactor and photos of the melt following the test (with corrections for heaters and electrodes).

Area of reactor floor (from CAD) = 1217 cm^2

Depth of Main Core and Foam (both over anode and not over anode, 7 in x 7 in) were averaged from CT data.

Main Core Depth (under anode) = 6.24 cm
Main Core Depth (not under anode) = 7.04 cm
Foam Depth (over anode) = 0.47 cm
Foam Depth (not over anode) = 1.21 cm

Volume Main Core = 8315 cm^3
Volume Foam = 1237 cm^3

Total mass was calculated from the densities of the previous slide (corrected for 4.1% Nb).

Total Mass of Remaining Melt = 20.8 kg
Mass Loss = 25.0196 kg – 20.8 kg = 4.2 kg

4.2 kg corresponds to 16.8% of regolith mass. However, we expect 0.564 kg O_2 max from electrolysis current measurements.

The theoretical limit of 0.564 kg O_2 corresponds to 2.3% of regolith mass converted to oxygen based on 25 kg.

Additional SEM/EDS of Core Slice

- SEM and EDS was also performed with a JEOL JSM-IT800.

Atom/Mol%													
Core Slice	O	C	Na	Mg	Al	Si	K	Ca	Ti	Fe	Nb	P	Mn
Average	54.89	0	2.15	2.19	12.43	14.81	0.42	5.1	0.63	0.46	10.09	0	0
Std. Dev.	3.18	0	0.6	2.09	3.02	3.95	0.07	2.72	0.35	0.13	7.32	0	0
ICN-LHT-1G Control	O	C	Na	Mg	Al	Si	K	Ca	Ti	Fe	Nb	P	Mn
Average	47.95	21.49	1.89	2.52	8.22	11.98	1.31	4.53	0.8	5.59	0	0.16	0.08
Std. Dev.	6.85	15.54	1.11	1.32	3.53	5	0.99	2.13	0.2	1.99	0	0.05	0

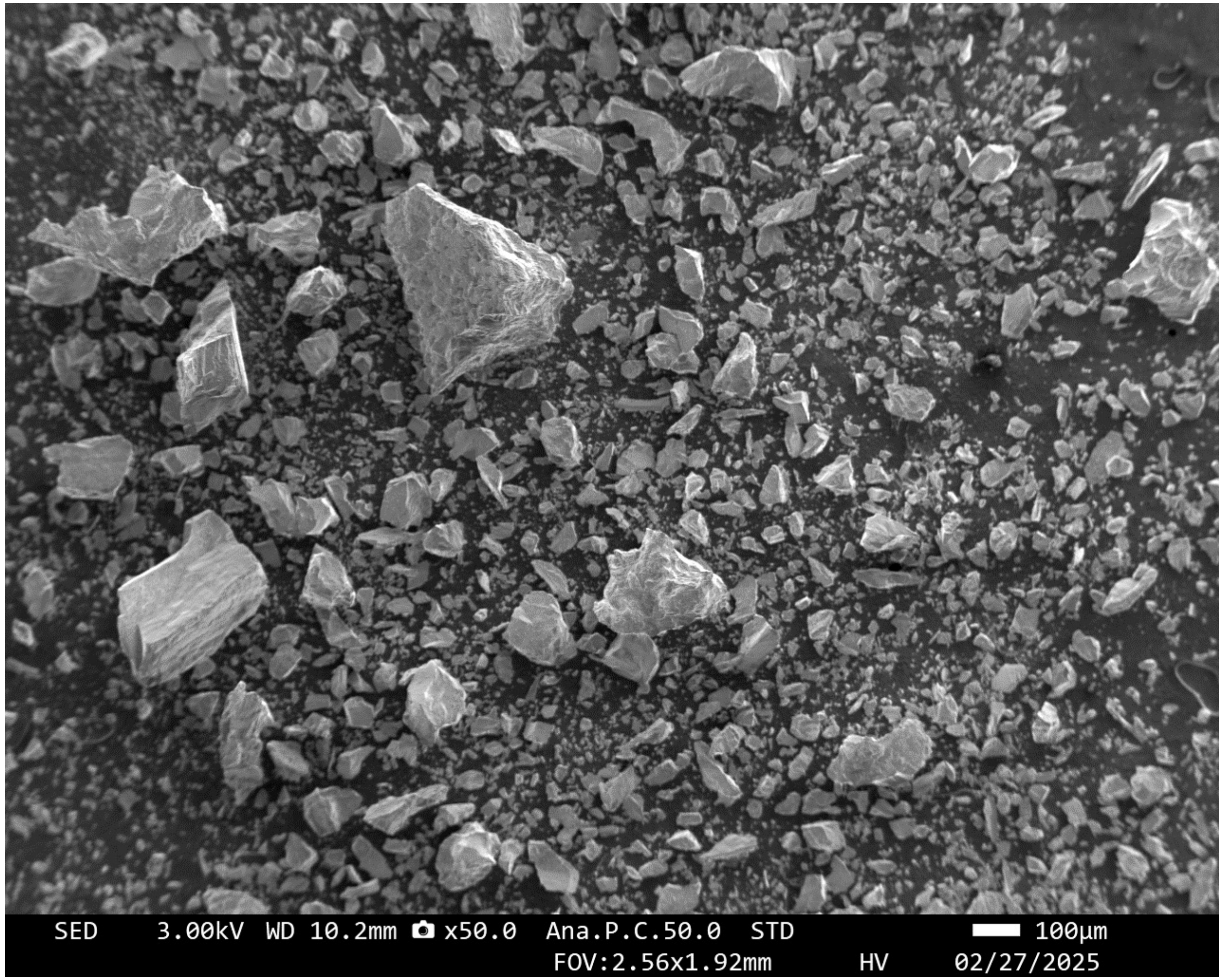
XRF
XPS

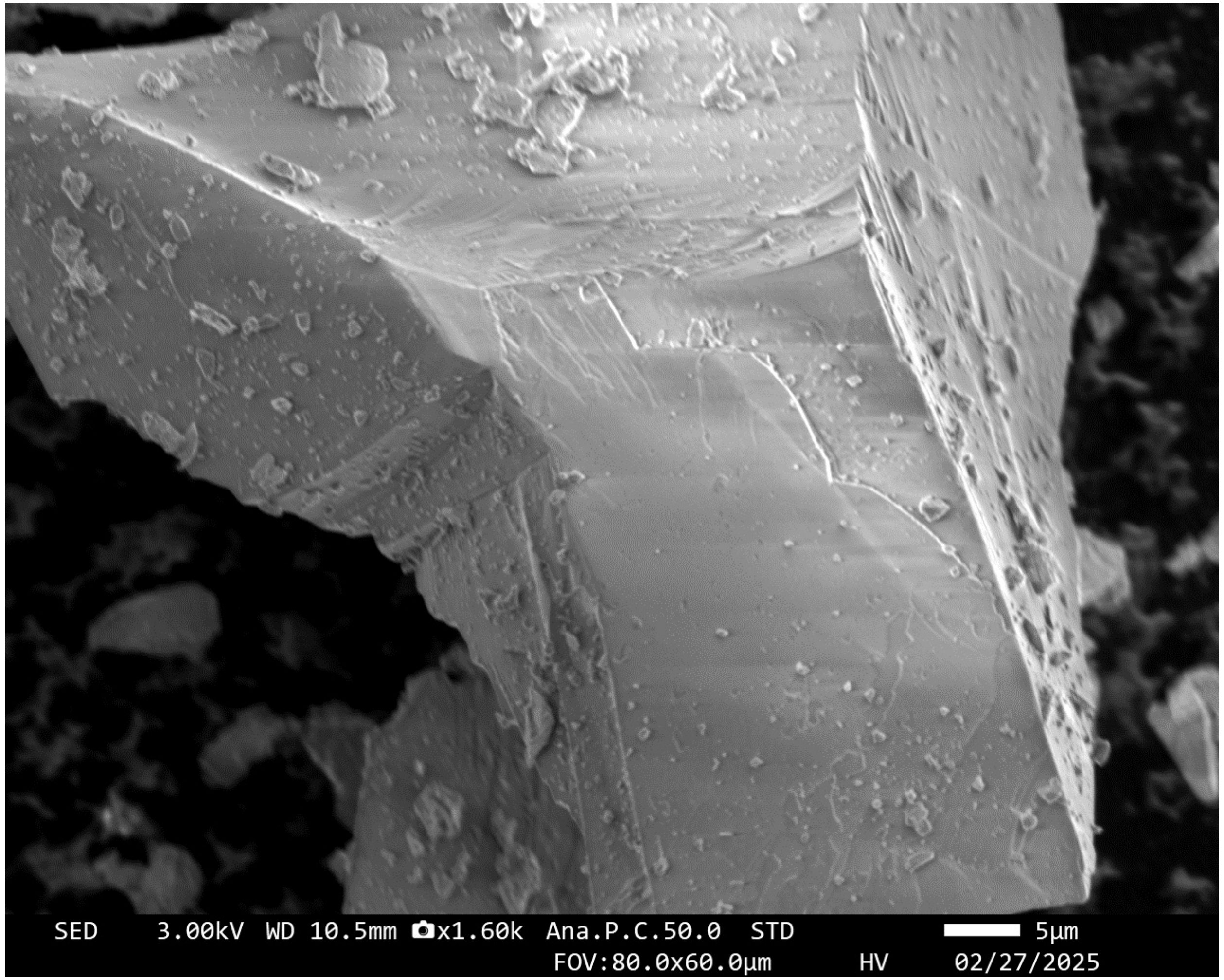
Possible Nb interference. Data not reported here.

[Ref: APL SAR 2021]

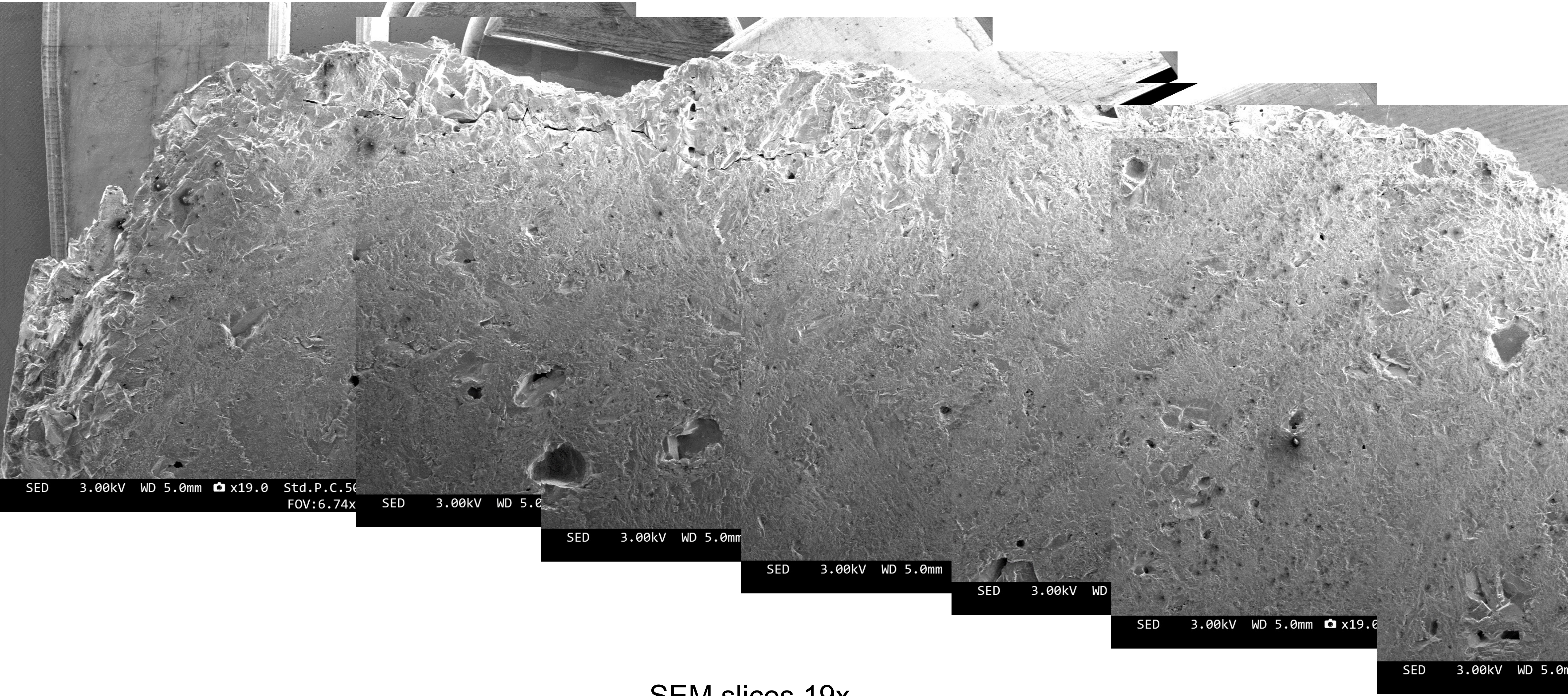
ICN-LHT-1G	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	TiO_2	FeO	MnO	P_2O_5
Wt%	2.60	1.90	22.90	48.0	0.0	15.9	2.90	5.70	0.0	0.0

SEM - ICN-LHT-1G – Control



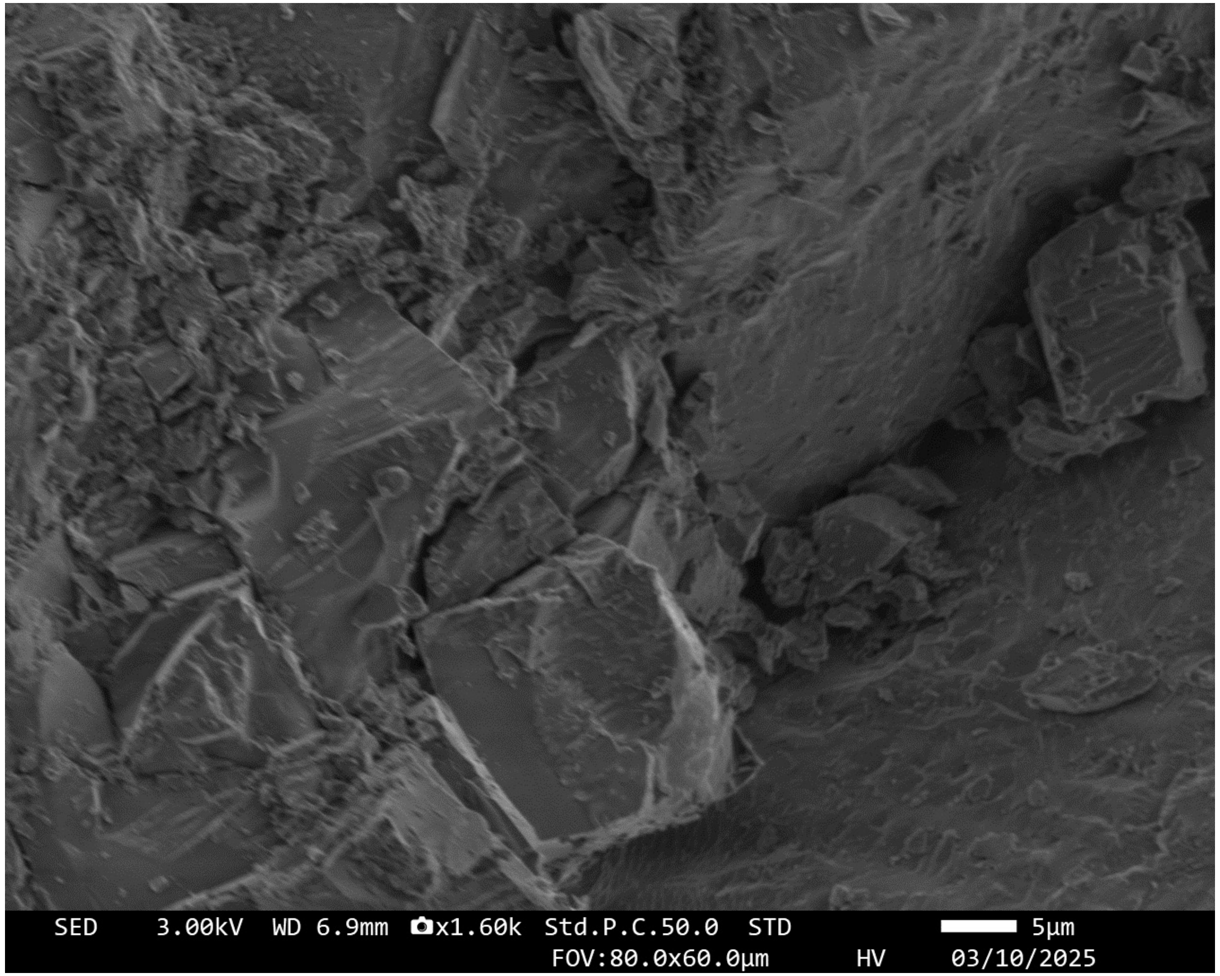


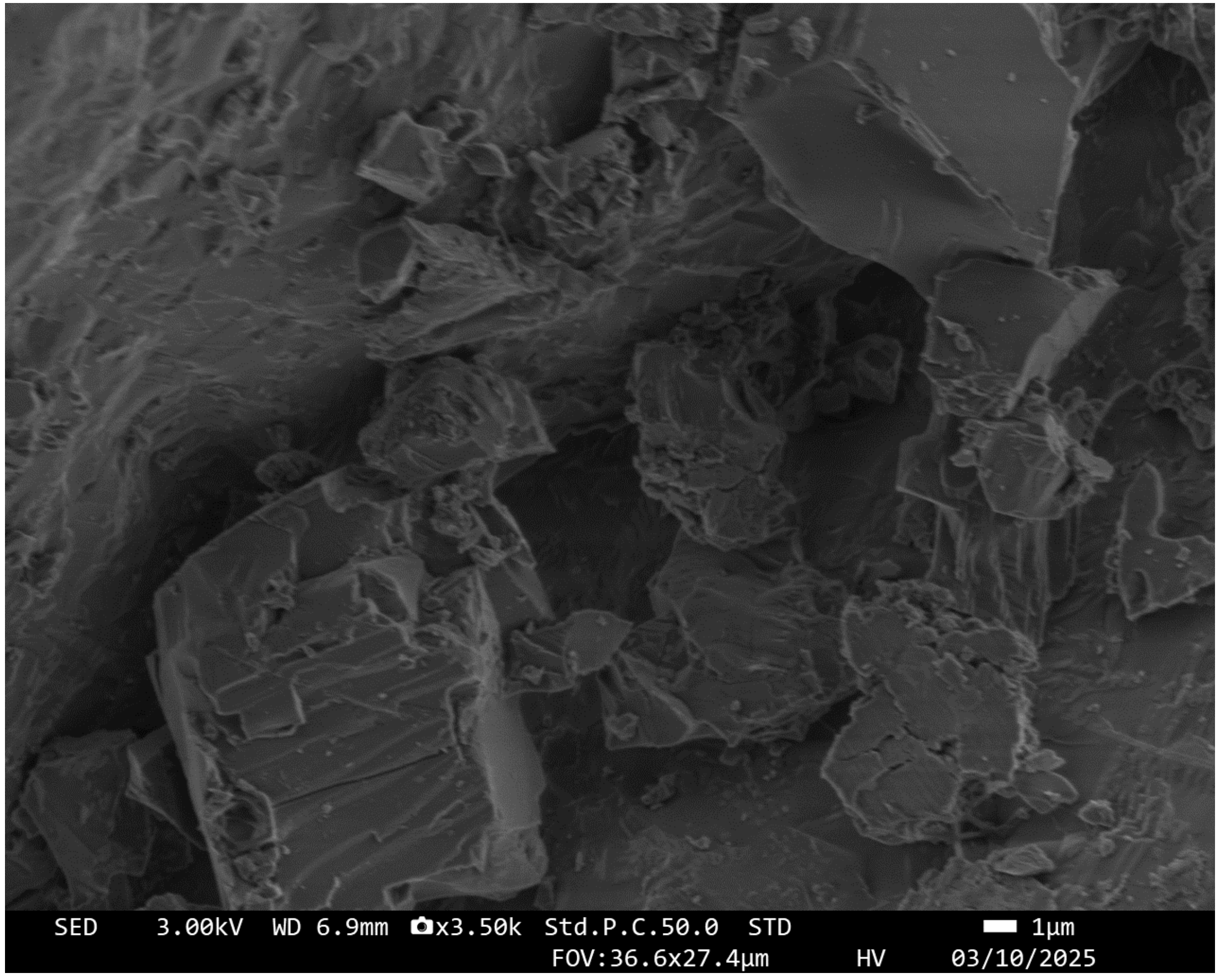
SEM – Core Slice Images



SEM slices 19x

SEM – Core Slice Images

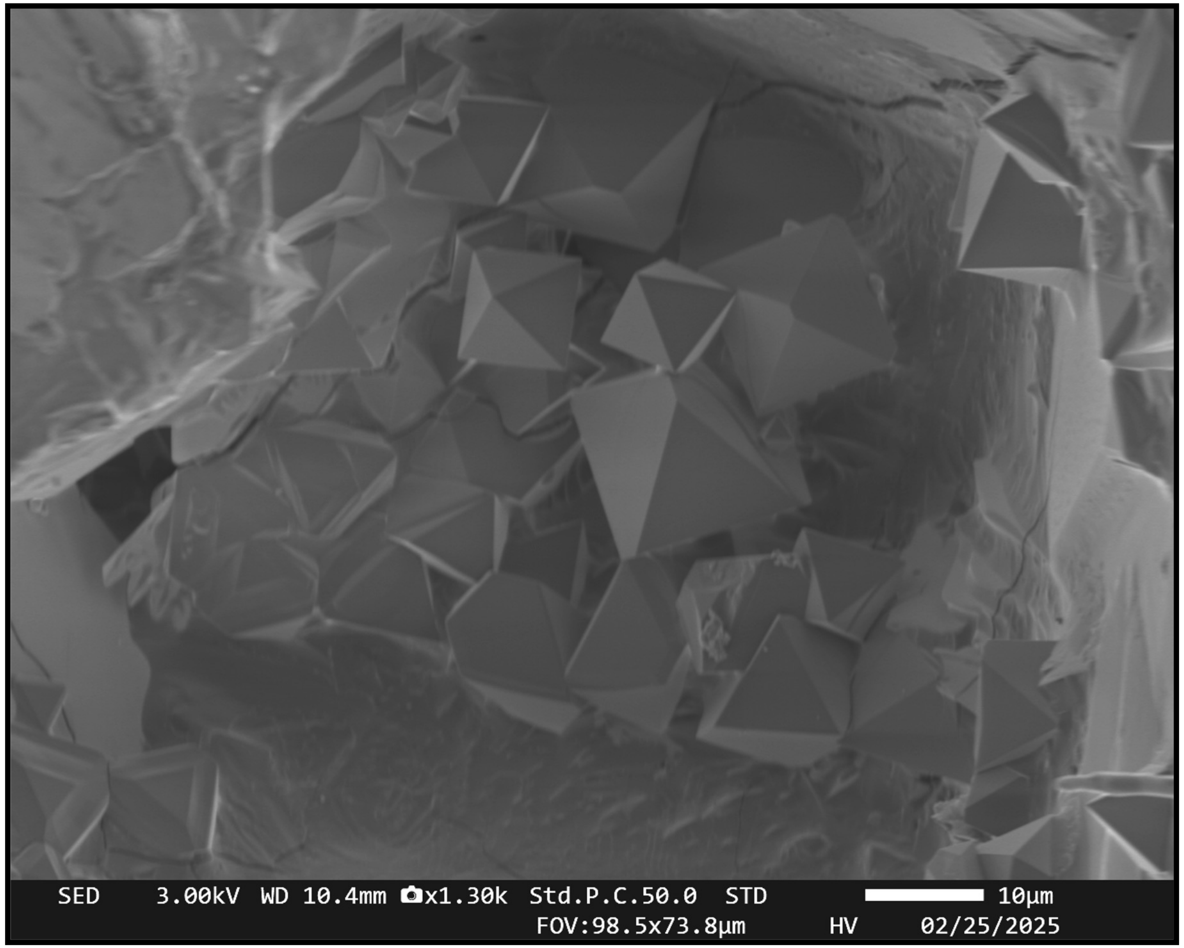


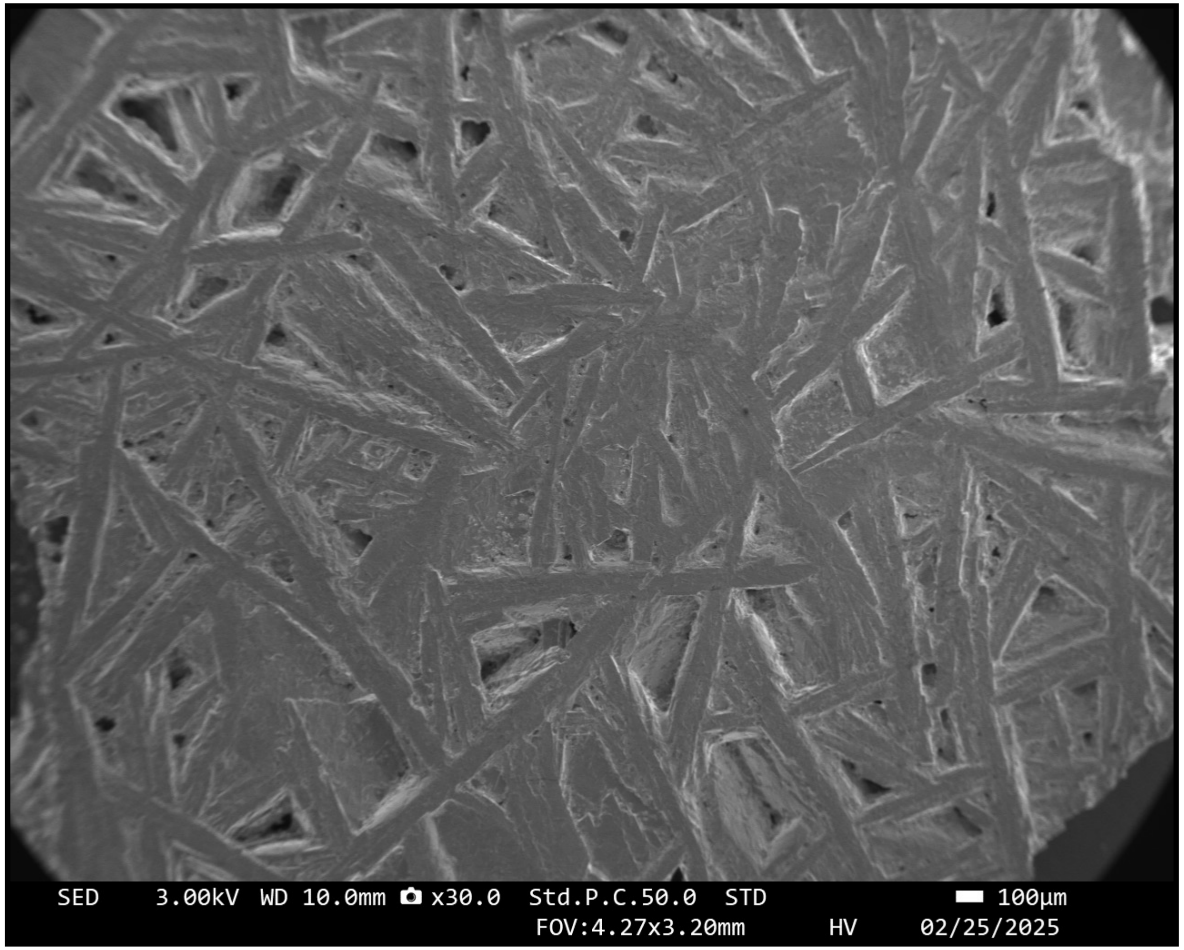


SEM – Not Core Slides

SEM images collected from areas around the surface of the reactor when the lid was removed.

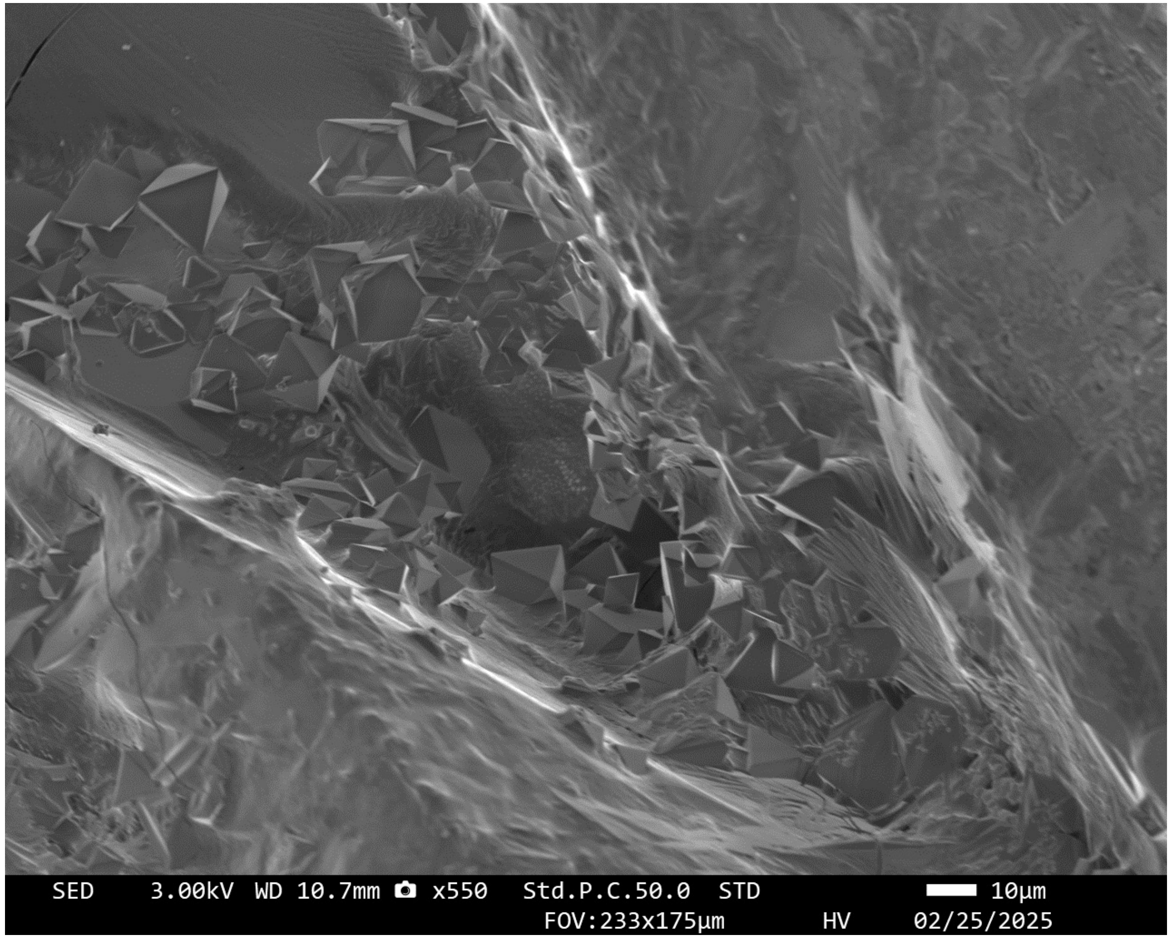
These areas had more surface exposure to cool and grow various precipitates / crystals.

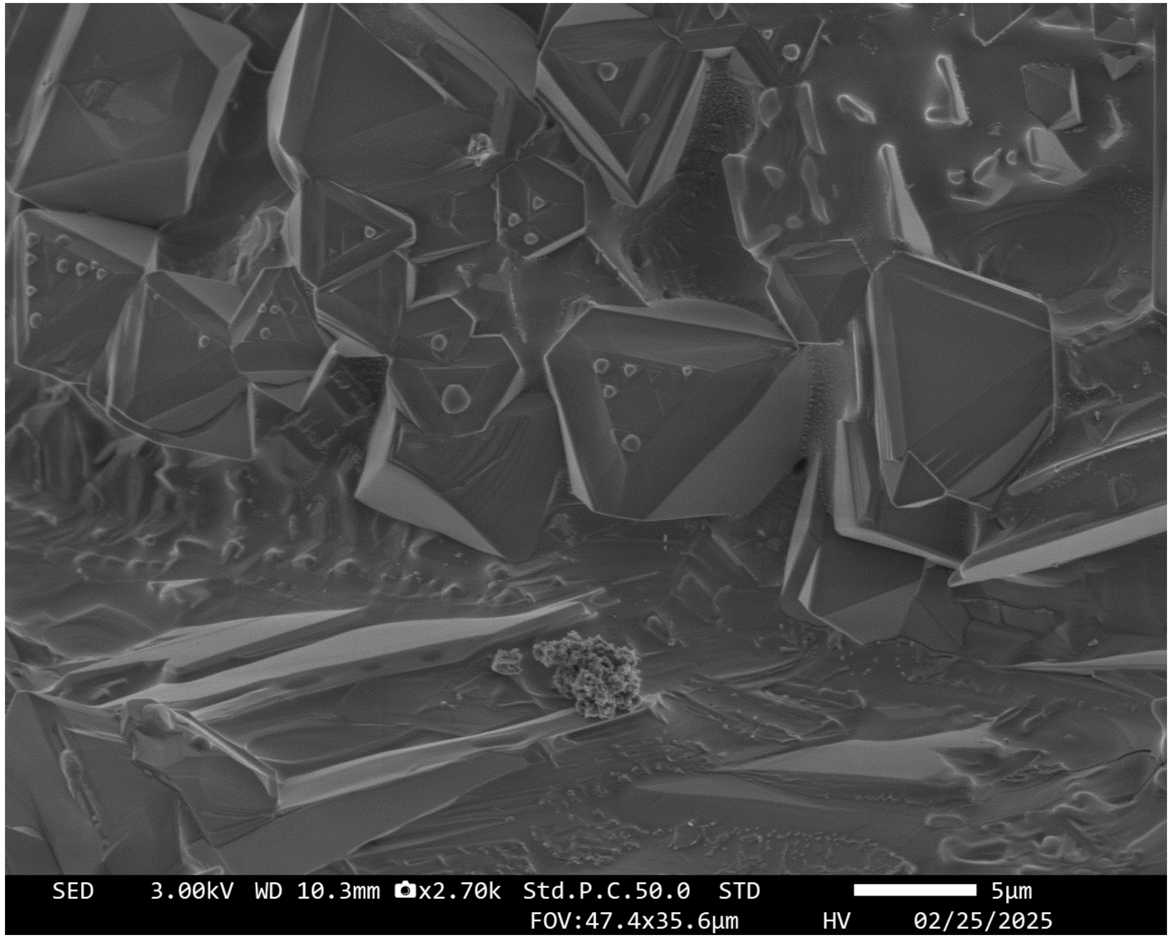




Fragments collected near the anode.

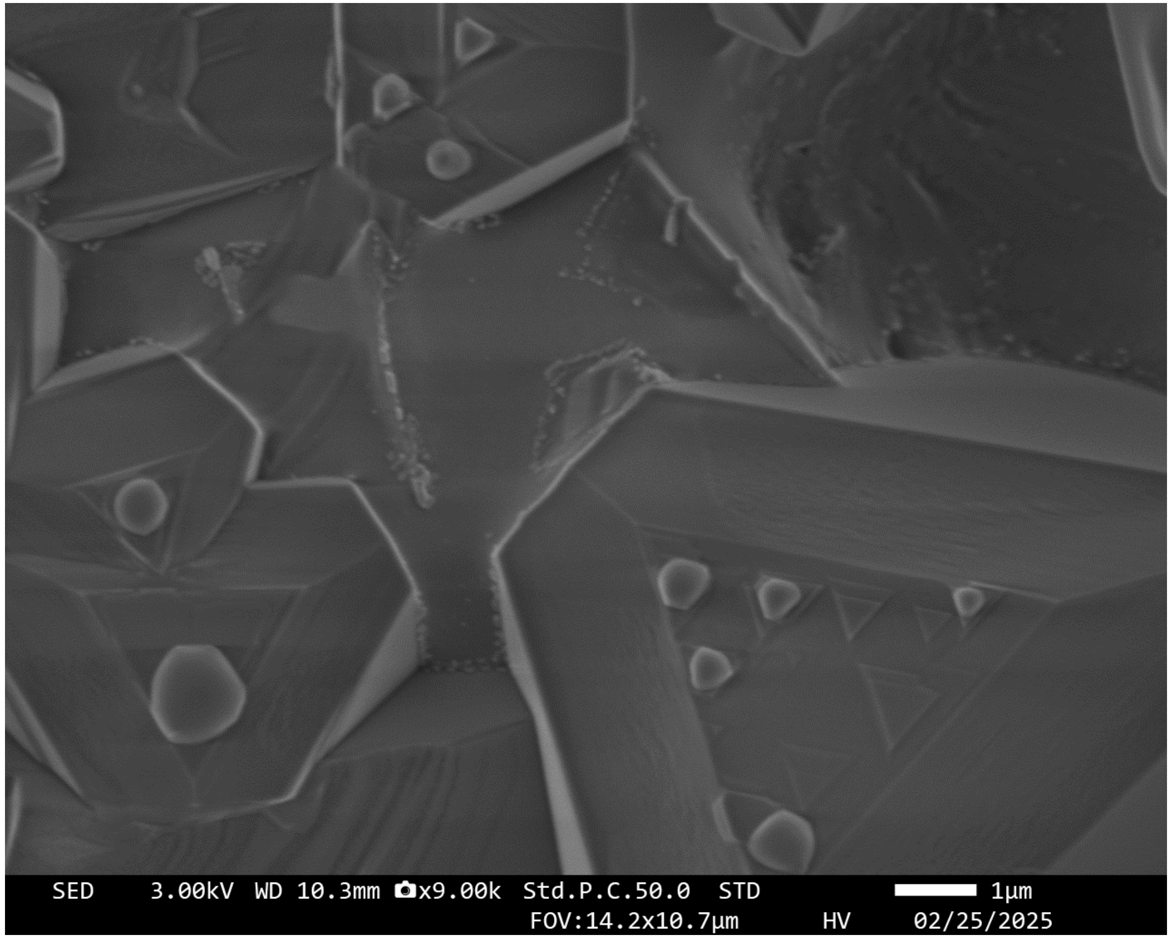
SEM – Other Fragments, Not Core Slices

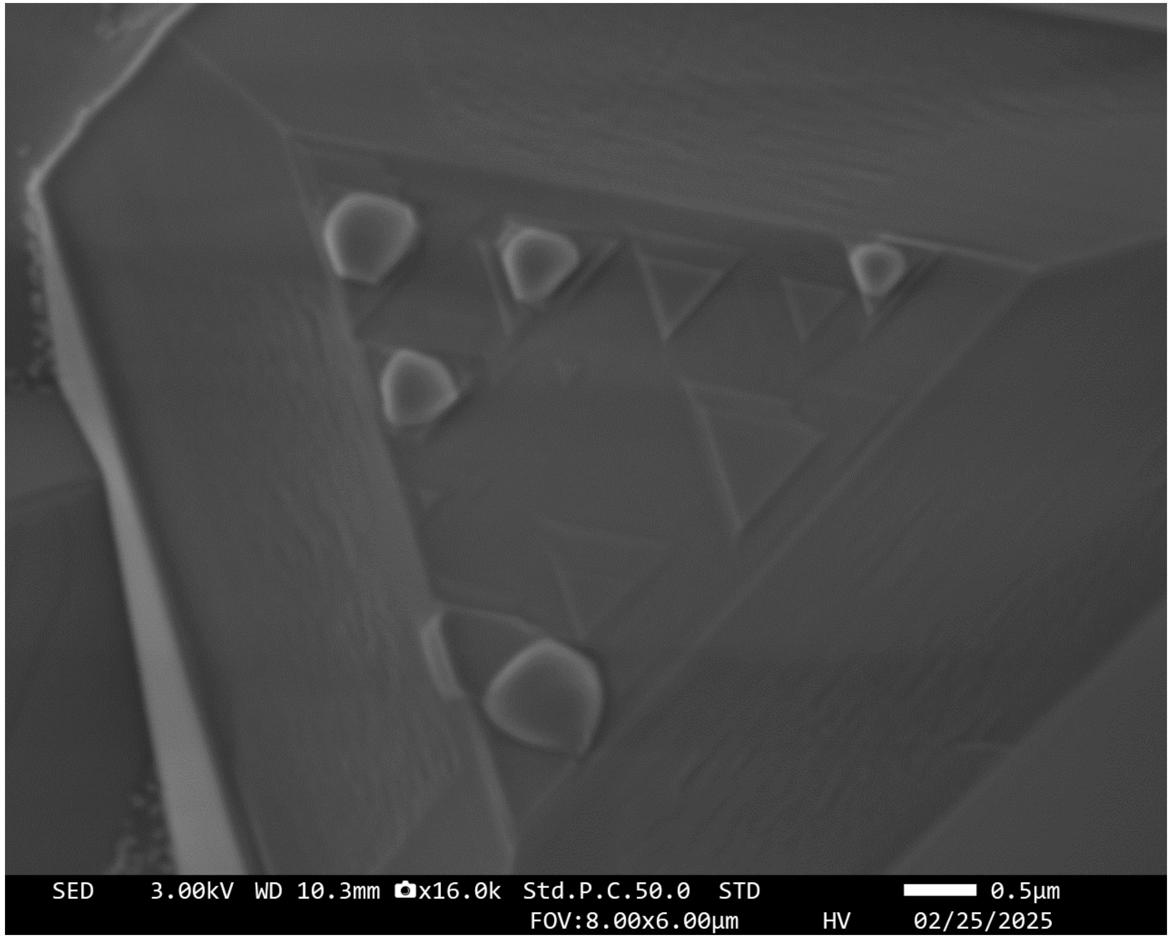




Fragments collected near the anode.

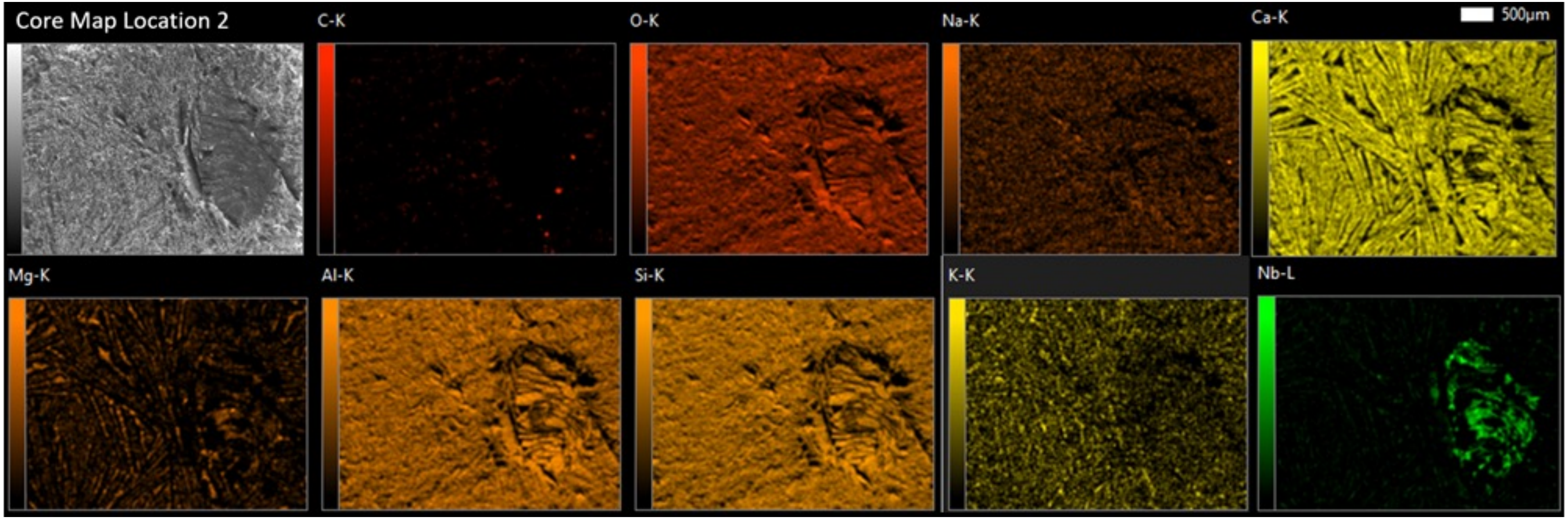
SEM – Not Core Slides





Fragments collected near the anode.

EDS – Mapping of Core Slice

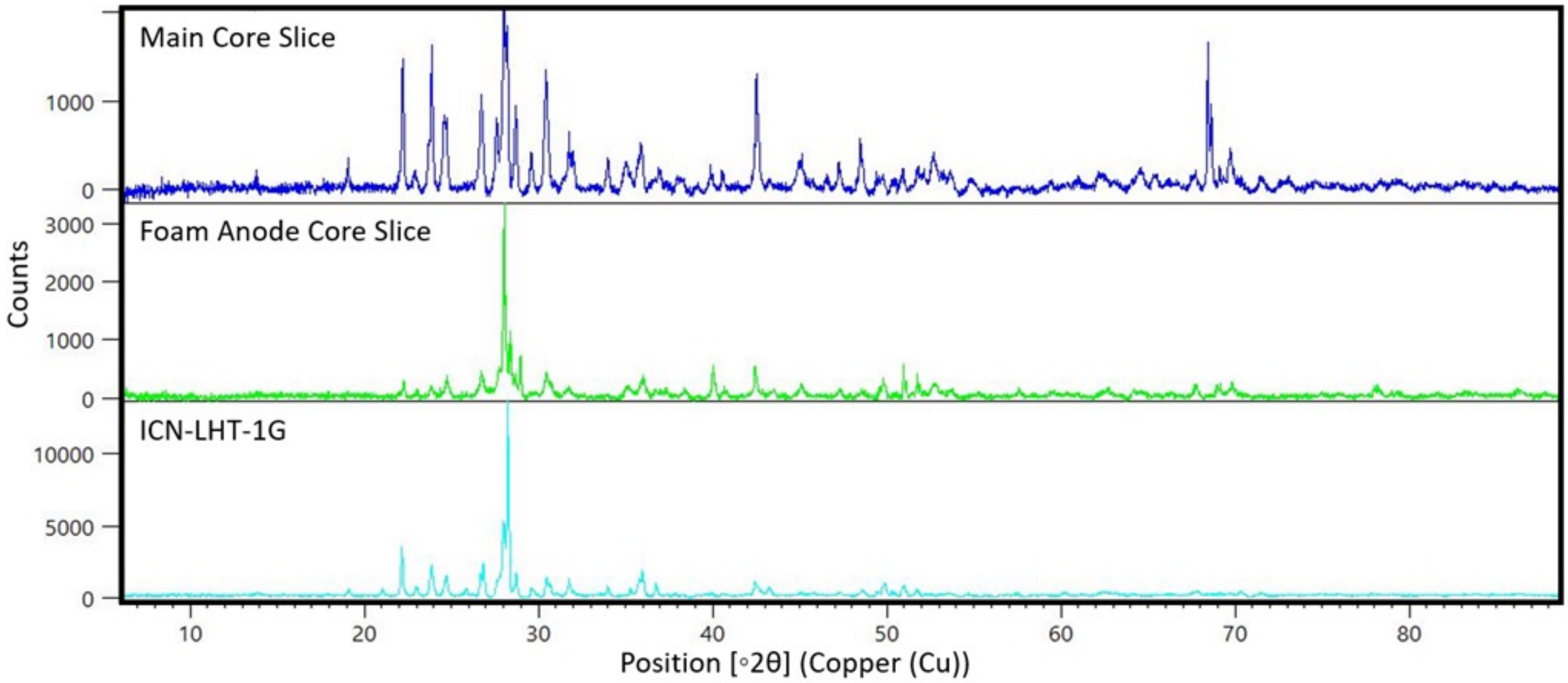


Elemental maps of the core slice, showing dendrites and mixed amorphous material with anode material.

XRD – Core vs Control Powder

XRD was performed on bulk material with a Malvern Panalytical Empyrean X-Ray Diffractometer.

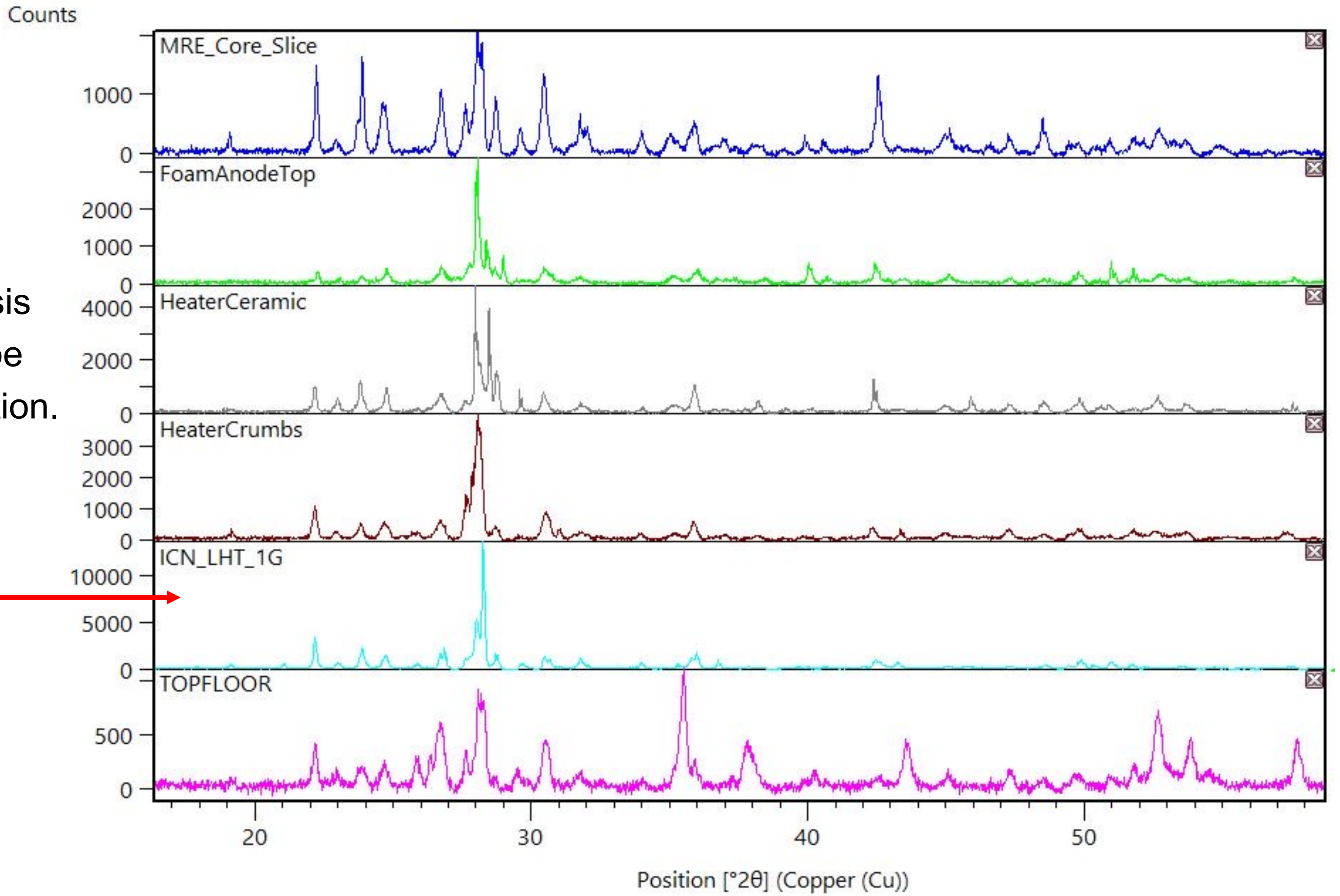
The main core slice displayed more crystalline formations with predominant sodium calcium aluminum silicate peaks, dominating in the anorthic crystal structure of the feldspar mineral class.



XRD – Various Areas, preliminary results

Further detailed XRD analysis and data interpretation will be prepared for journal publication.

Control LHT-1G Powder



Summary

- Large scale electrolysis was achieved for MRE operations in a vacuum environment using 25 kg of ICN-LHT-1G regolith simulant. A core sample of the solidified melt was successfully removed after cool-down phase.
- Successful oxygen production was achieved during the test. Measured rate: 0.067 kg/h, 0.3 mol/kW-hr.
- Voids are present throughout with crystal structures formed in the voids. CT was used to determine macro-porosity of approximately 14.84%
- EDS observations: Non-homogeneous properties with mixed elemental composition. Anode degradation was observed throughout regolith magma in core slice.
- XRD observations: The main core slice became more crystalline relative to the regolith, possibly due to local crystallization upon solidification.
- XPS was performed with a Thermo Scientific NEXSA G2. Due to Nb presence in the core from the anode material, it is believed that x-rays were absorbed and the data were inconclusive.

Acknowledgements

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•KSC NASA Mechanical Analysis Lab (MAL).

•NASA Investigative Chemistry Lab (NICL).

•Granular Mechanics and Regolith Operations Lab (GMRO).

•Applied Chemistry Laboratories (ACL).

References:

[1] Standish (2010) Design of a Molten Materials Handling Device for Support of Molten Regolith Electrolysis.

[2] S. S. Schreiner, (2015) Molten Regolith Electrolysis reactor modeling and optimization of in-situ resource utilization systems.

[3] A. Slabic et al., (2024) Lunar Regolith Simulant User’s Guide Revision A.

Members & Stakeholders

KSC MRE Team
- **Jaime Toro, PI, Data Acquisition, NE-L1**
- **Annie Meier, PM, Chemical Engineer, UB-E**
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- **Mark Hinkel, PM, Lunar Resources, Inc.**
- **Rabi Ebrahim, Lunar Resources, Inc.**

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- **Christopher Kuhl, Program Chief Engineer, GCD, STMD**
- **Duane Pettit, Program Safety Officer, GCD, STMD**
- **David Moore, Program Manager, GCD, STMD**
- **Gerald (Jerry) Sanders, ISRU System Capability Lead, STMD**

KSC Chief Engineers/Review Board
- **Marty Grashik, R&T Chief Engineer**

Questions

?

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Backup

Technical Performance

Project Plan KPPs

Performance Parameter	Units	Threshold Value	Goal
KPP 1: Produced Oxygen Mass (as percent of regolith melt mass). Test duration target of 14 hours at target current.	Weight %	5.7	10
KPP 2: Average Oxygen Production Rate	kg/h	0.1	0.2
KPP 3: Oxygen Production Energy Efficiency (During electrolysis)	Mols O2/kW-hr	0.6	1.0
KPP 4: Energy Efficiency for Melt Phase (pre-electrolysis melt phase)	kJ/kg	2000	1500

Recommended KPPs

Performance Parameter	Units	Threshold Value	Goal	Measured
KPP 1: Produced Oxygen Mass (as percent of regolith melt mass). Test duration target **of 9 hours at 210 Amps**	Weight %	1.54	3.15	2.3
KPP 2: Average Oxygen Production Rate	kg/h	0.04	0.08	0.07
KPP 3: Oxygen Production Energy Efficiency (During electrolysis)	Mols O2/kW-hr	0.6	1.0	0.3
KPP 4: Energy Efficiency for Melt Phase (pre-electrolysis melt phase)	kJ/kg	3,100	4,428	11,877@1200C 24,692@1300C

Project Plan KPPs:
- Test parameters
- Internal LR formulations

Test Execution Impacts on KPPs:
- Test modifications:
  - Power, Oxygen quantification, Off-nominal events
- Data quantification and qualitative information:
  - Overall Oxygen data: off nominal situations (liquid intrusion/ leaks), changed measured impacted O2 values.
  - Steady state oxygen data
- Extrapolation
  - Oxygen based on steady state
  - Reduced mass based on core
  - Energy based on temperature regions.

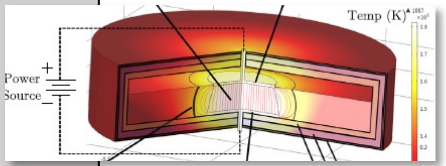
Recommended KPPs consider:
- Methodology of derivation – KSC
- As-run power configuration
- Extrapolations
- Linear assumptions

Molten Regolith Electrolysis History

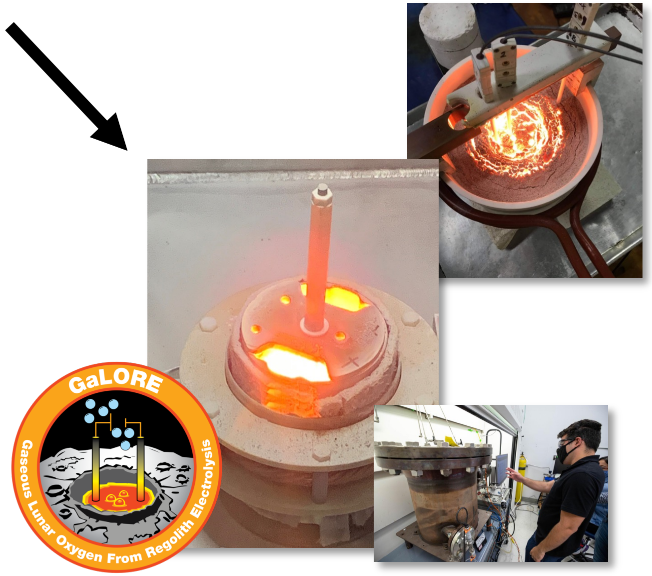
1990: A case for MRE is reported [1]

2009: Electrode materials are demonstrated for MRE in a lab environment [2]

2015: Parametric modeling investigations into MRE [3]

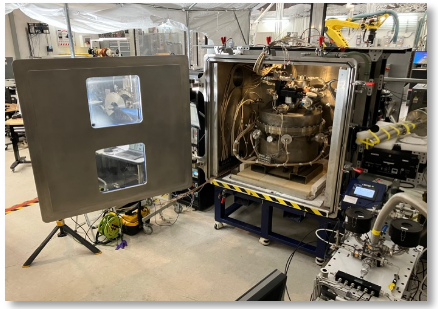


Parametric Sizing



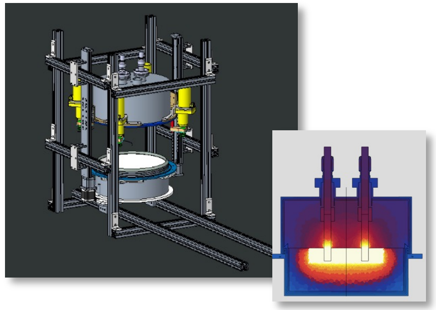
2023: GaLORE ECI develops 3 regolith melting methods and demonstrates cold-wall reactor at KSC

Relevant environment Melting technology & oxygen quantification



2024 MRE GCD: demonstrates a large-scale reactor in lunar like conditions.

Cold-wall & Automation



2025 FORGE IRAD: demonstrating batch-based for a continuous production reactor design.

Commercial Partner Infusion

2023+ KSC led infusion:
Partnerships to provide system expertise

MUREP Grants and independent research

SBIR/STTR technical oversite

Produced O2 & additional product makeup

Continuous processing

2025+ C3-PO: investigate *purification*, cooling, and collecting MRE IRSU produced O2

*While MRE is a 1-stage chemical reaction that provides both oxygen and valuable metals, **current technology gaps exist for purification/storage of O2, metals extraction, and further refinement of a design for batch-based or continuous function of the reactor.***

[1] Colson, Russell O., and Larry A. Haskin. "Lunar oxygen and metal for use in near-earth space: Magma electrolysis." *Arizona Univ., NASA Space Engineering Research Center for Utilization of Local Planetary Resources* (1990).
[2] Sibille, Laurent, et al. "Recent advances in scale-up development of molten regolith electrolysis for oxygen production in support of a lunar base." 47th AIAA Aerospace Sciences Meeting including The New Horizons Forum and Aerospace Exposition. 2009.
[3] Schreiner, Samuel Steven. Molten Regolith Electrolysis reactor modeling and optimization of in-situ resource utilization systems. Diss. Massachusetts Institute of Technology, 2015.

MRE Tech Maturation Project Overview

Technology Development Needs Addressed by Project		
Stakeholder (ISRU STMD; ESDMD)	This project aims to demonstrate In-situ production of O2 and metals with lunar regolith.	STARPort : 564 - Oxygen Extraction from Lunar regolith 566 - Metal extraction from regolith

<u>STMD Civil Shortfalls Addressed by MRE GCD:</u>

- ✓ **1580:** Extraction and separation of oxygen from extraterrestrial minerals (Ranked 68)
- ✓ **1581**: Extraction and separation of extraterrestrial atmospheric resources and gaseous products/reactants (Ranked 107)
- ✓ **1582**: Extraction and separation of metals/metalloids from extraterrestrial minerals (Ranked 110)
- ✓ **1583**: Produce propellants and mission consumables from extract In-Situ Resources (Ranked 79)
- ✓ **1593**: Lunar surface power generation from ISRU derived resources (Ranked 178)

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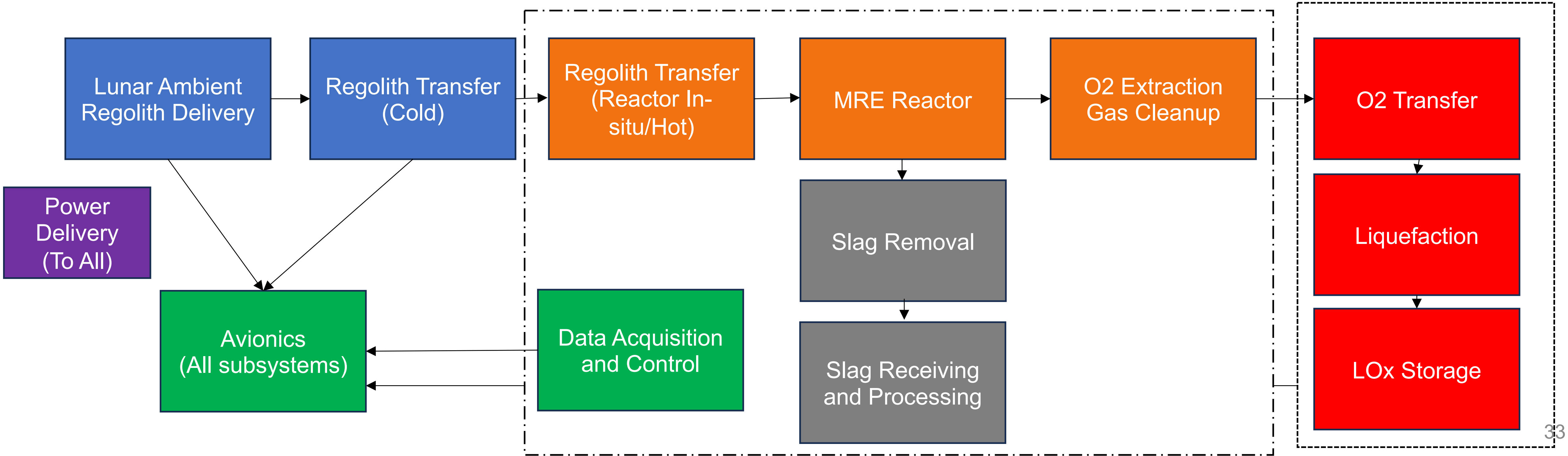
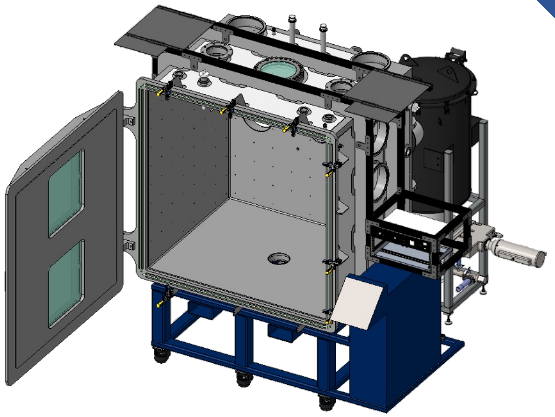
MRE PM/SE Approach & TRL Discussion

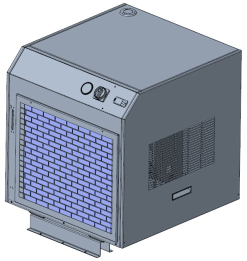
- For the entire MRE System (including reactor, other subsystems and components), development is required under NPR 7120.8A Research and Technology Program and Project Management Requirements
- To move to flight, the entire MRE System must advance to 7120.5 (with process tailoring to increase risk posture).



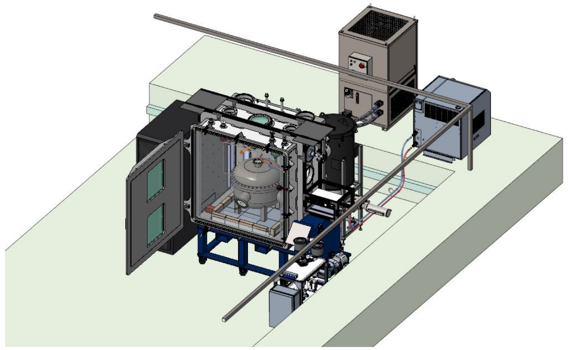
Hot Reactor Proving Grounds - ASSIST

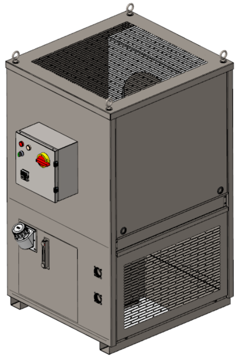


ASSIST Environment Chamber
3E-6 Torr Minimum Pressure
1.4 x 1.1 x 1.4 m test volume

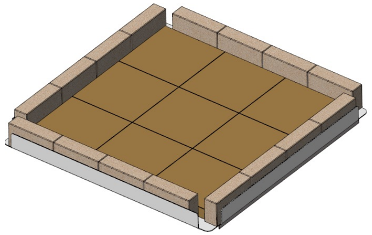


ASSIST Exterior Wall Cooling (7kw)





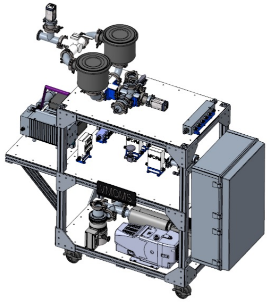
Reactor Interior Oil Cooling (10kw)



Ceramic Firebrick Pallet

3 Power Supplies 10-15kW ea.

DAQ Capabilities
- Temperature
- Cameras
- Pressure



O2 Monitoring