Ultra-High Vacuum Thermal and Carbothermal Reduction of Lunar Regolith Simulants for Oxygen and Construction Materials

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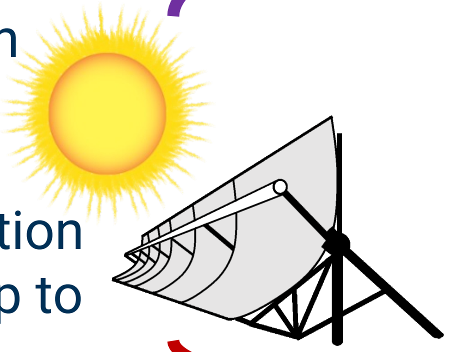
Outline

- Background and Motivation
- Methodologies
- Results
  - Material characterization
- Summary and conclusions

Background and Motivation

Thermochemical processing of lunar regolith to produce oxygen and construction materials for ISRU to advance space exploration efforts

- No atmospheric attenuation
- Long lunar day
- Concentration capacity up to 5000 suns



Solar Thermal Reduction

Lunar Regolith

Metals/ Metalloids + **Oxygen**

Valuable as construction materials

Valuable as rocket propellant and life support

Solar Carbothermal Reduction

Carbon

CO Recycling

Found on Moon as methane or human waste upon lunar colonization

- Facilitates high temperature separation of oxygen from oxides
- Lower reaction onset temperatures

Lunar Regolith Simulants

JSC-1A

- Simulating Apollo sample 14163
- Oxides (weight %):
  - SiO_2 – 47.4%
  - Al_2O_3 – 16.1%
  - Fe_2O_3 – 11.4%

LMS-1

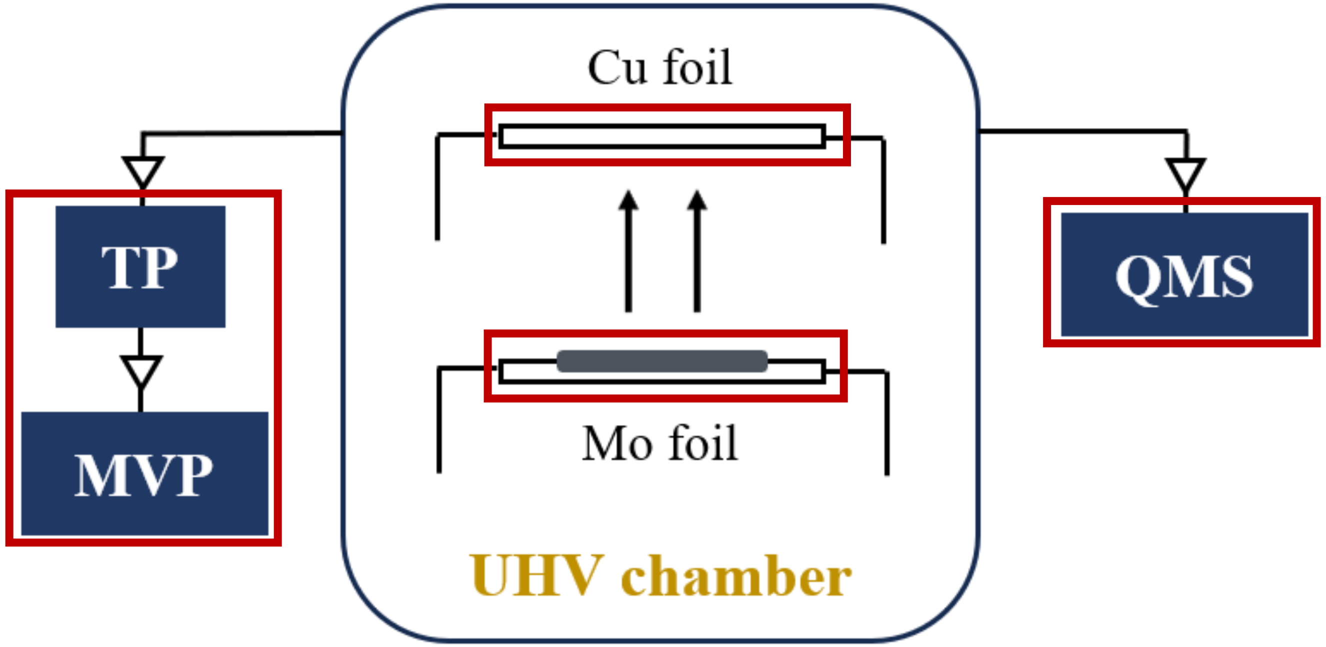
- Generic mare
- Oxides (weight %):
  - SiO_2 – 46.9%
  - MgO – 16.8%
  - Al_2O_3 – 12.4%

LHS-1

- Generic highland
  Oxides (weight %):
  - SiO_2 – 51.2%
  - Al_2O_3 – 26.6%
  - CaO – 12.8%

UHV Experiment Methodology

- Continuously evacuated via turbomolecular in series with mechanical vacuum pump to maintain ~10^{-12} bar, imitating low-pressure lunar surface conditions
- Sample of ~5 mg spread on an Mo foil, and Cu foil placed ~3 mm above the sample for vapor depositions
- Quadrupole mass spectrometry (QMS) attached to UHV chamber for measuring gas evolution (O_2 or CO)



UHV Experiment Methodology: Direct Thermal

- Two experiments conducted consecutively using the same sample for each simulant
- Thermodynamic analyses conducted at ~10^{-12} bar to determine theoretical operating temperatures for targeted metal production
- Si(g) was not targeted due to requirement of T ≥ 2000 °C for production
- SiO(g) was targeted instead as it is an intermediary in Si production
- Isotherm duration was shortened at higher *P* runs to avoid Mo sample holder deterioration

Simulant	*P*, W	*T*, °C		Targeted metal	Isotherm duration, min
		Target	Measured		
LMS-1	613.2	1170	1182-1195	Mg(g), SiO(g)	60
	730.0	1380	1234-1245	Al(g), SiO(g)	30
LHS-1	835.9	1300	1304-1313	Ca(g), SiO(g)	30
	985.5	1500	1484-1498	Al(g), SiO(g)	5

UHV Experiment Methodology: Carbothermal

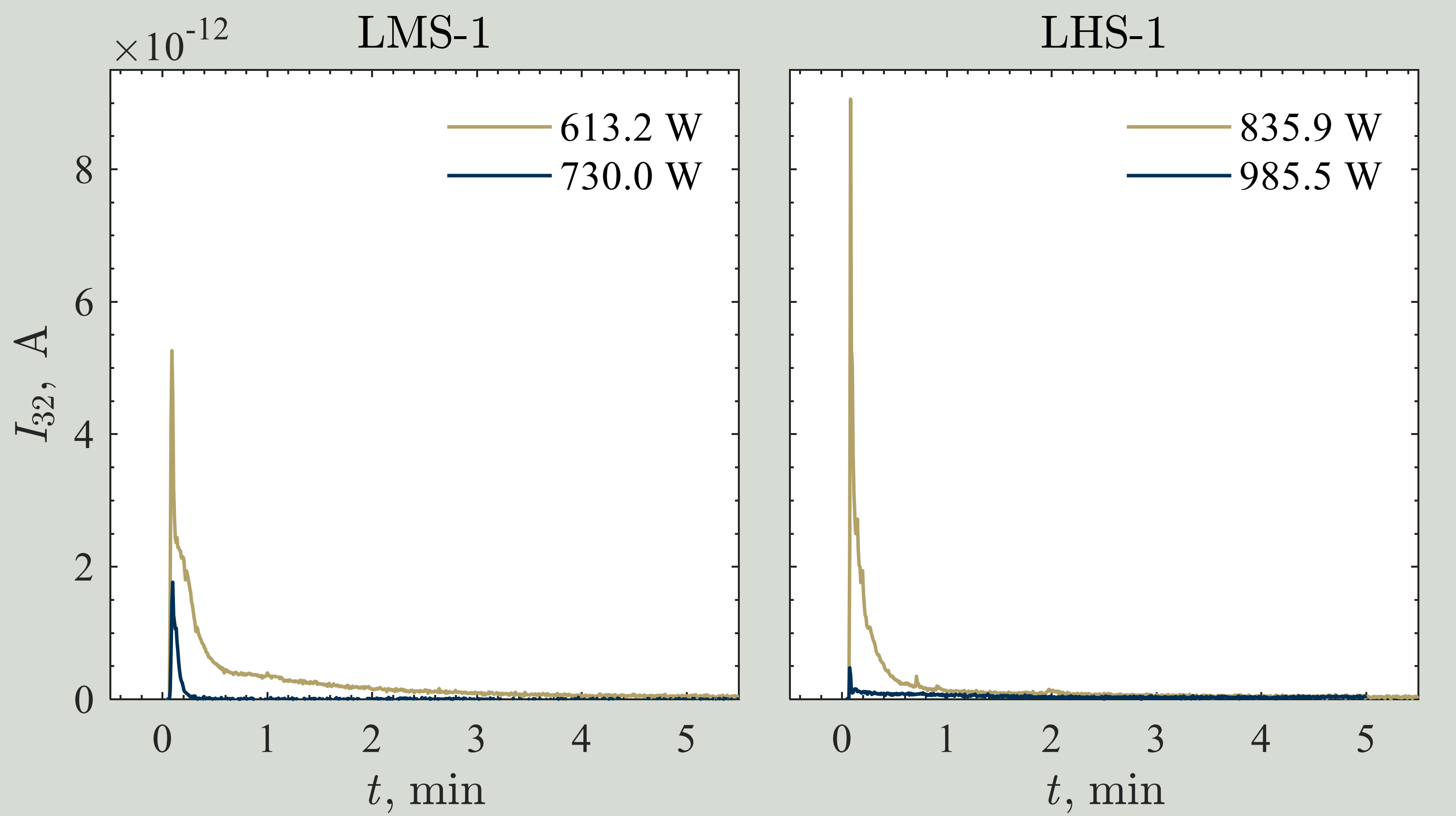
- Simulants were mechanically mixed with pyrolyzed activated carbon at C to simulant mass ratio of 0.5. This ratio represents larger amounts of C than theoretically needed to reduce all oxides in the simulants.
- Two experiments conducted consecutively using the same sample for each simulant mixture
- $T \geq 1200$ °C and ≥ 1400 °C were chosen since previous study has shown that $T > 1200$ °C is needed for vapor production

Simulant mixture	P, W	T, °C		Isotherm duration, min
		Target	Measured	
JSC-1A + C	766.5	≥1200	1243-1256	60
	912.5	≥1400	1415-1432	30
LHS-1 + C	803.0	≥1200	1366-1380	60
	876.0	≥1400	1455-1458	30

Direct Thermal Reduction

- O_2(g) evolution from sample during experiments was evidenced by the QMS at 32 amu
- A prominent peak at t ≤ 0.5 min was observed with no significant changes in I_{32} for t > 4 min, indicative of rapid thermal reductions
- Smaller amounts of O_2(g) evolved during the second experiments because the reacted sample from first experiments were used causing smaller sample size

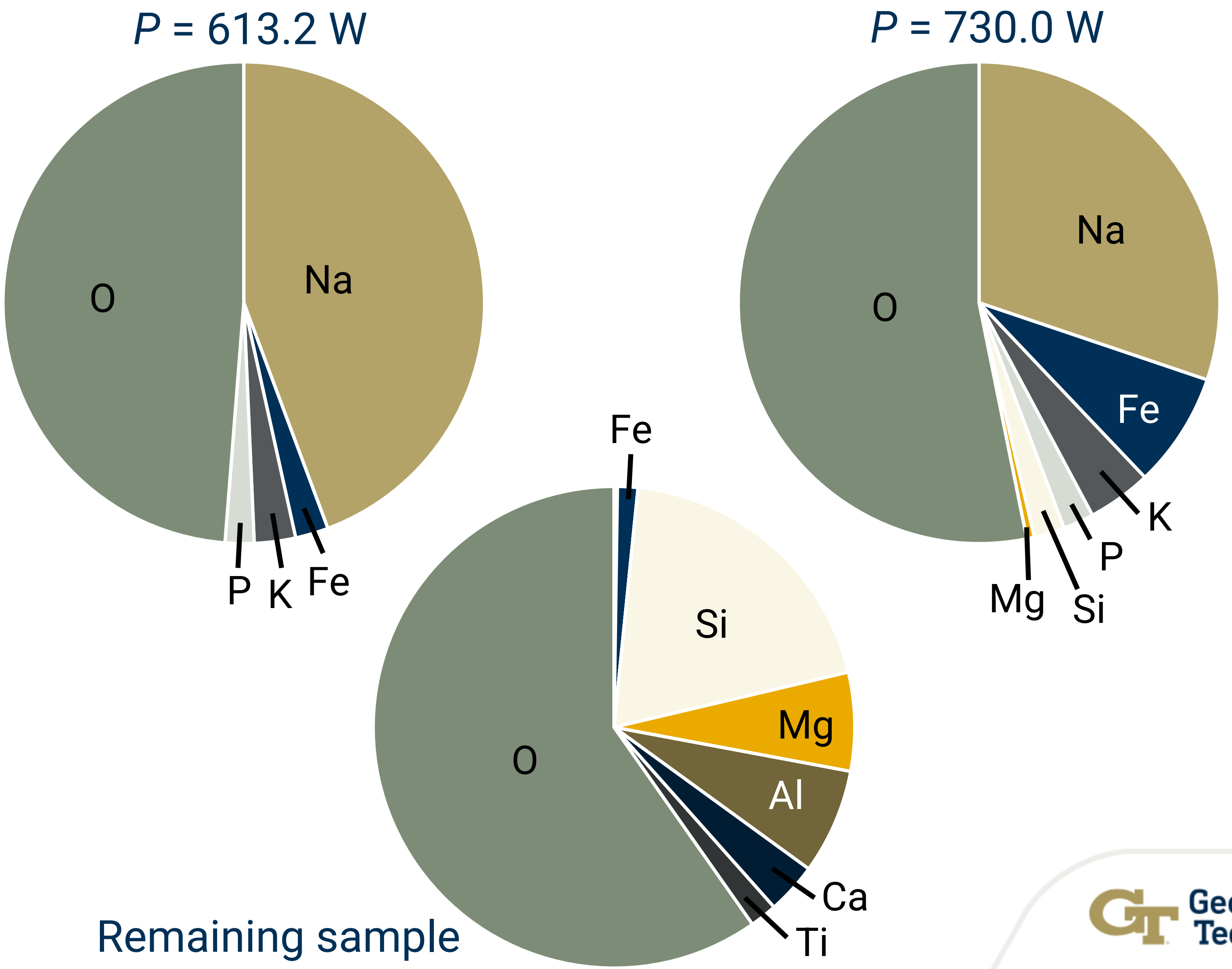
Ionic current of amu 32 as measured by QMS



Direct Thermal Reduction: Material Characterization

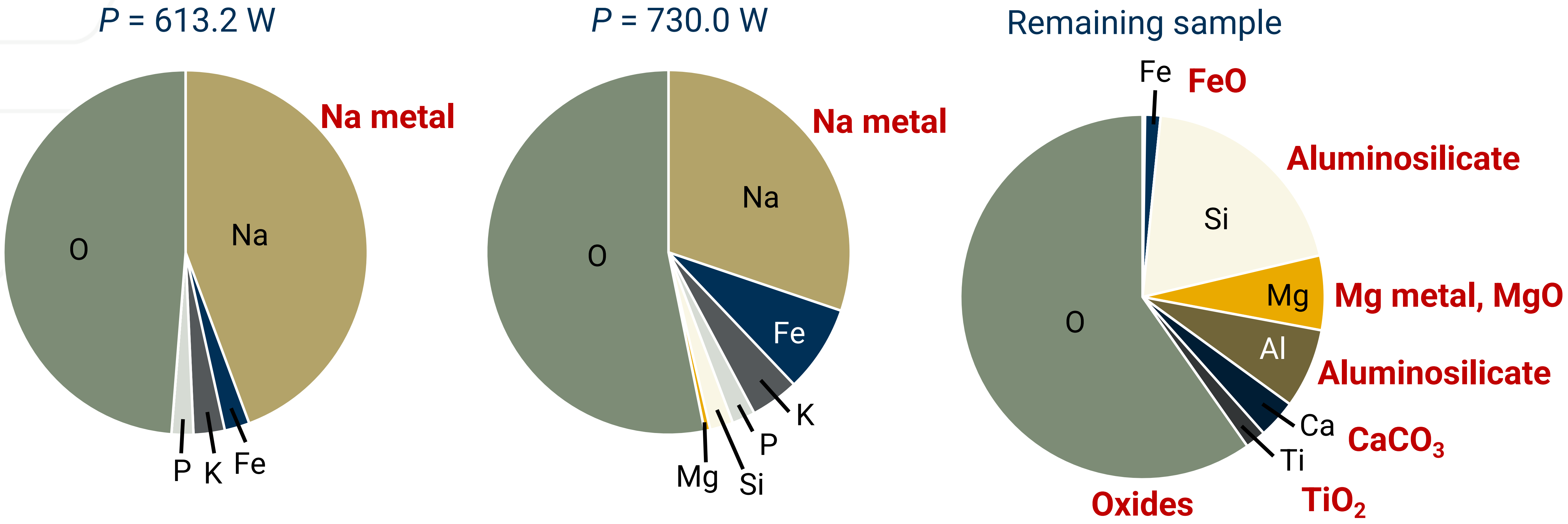
LMS-1 – EDS

- Depositions were evenly distributed on all foils
- Na, Fe, K, and P evolved during both experiments suggesting separation possibility at reasonable temperatures
- Si and Mg evolved during the second experiment and Al, Ca, and Ti remained in sample upon experimentation evidencing requirement of elevated temperatures



Direct Thermal Reduction: Material Characterization

LMS-1 – XPS

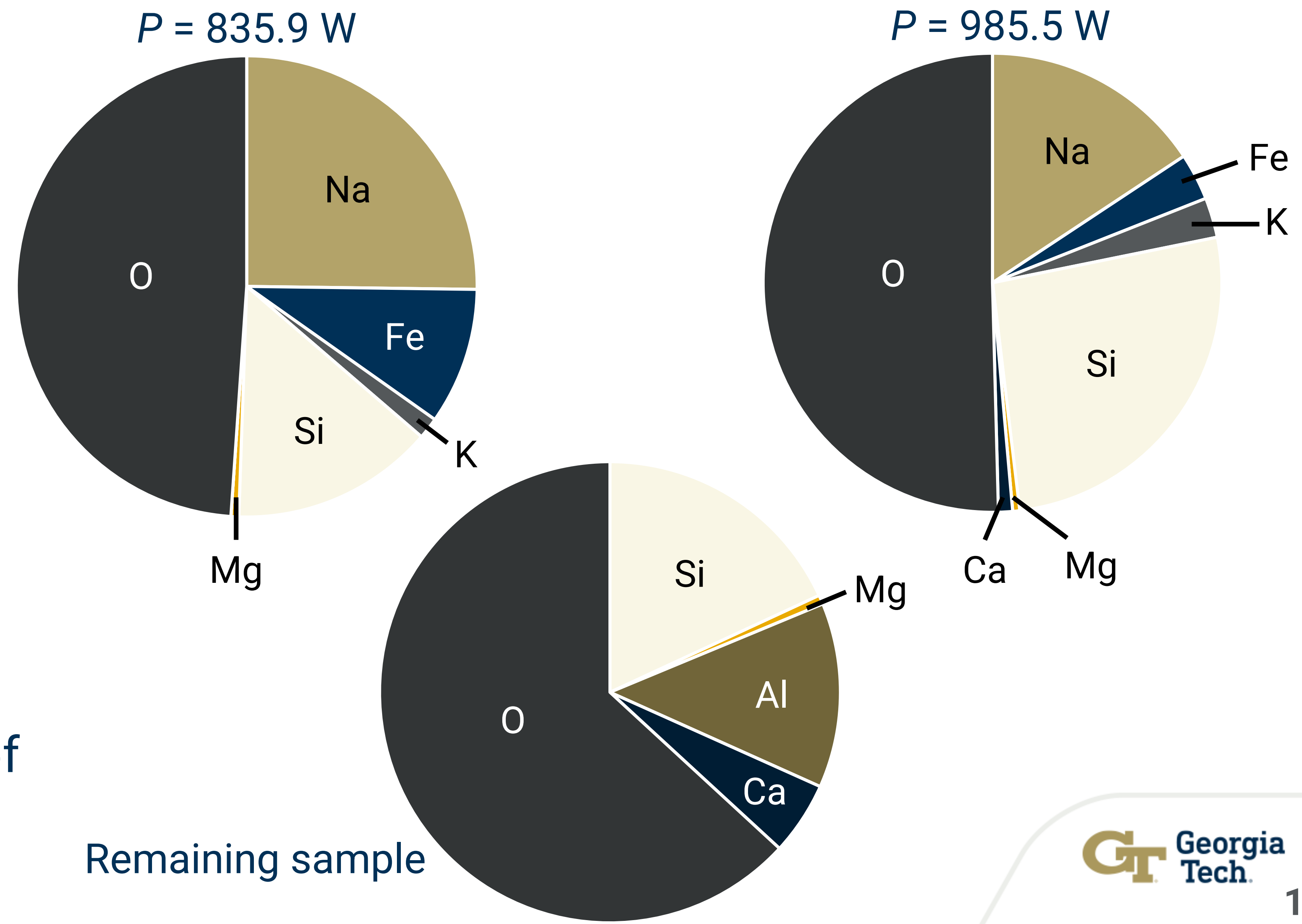


- Depth of analysis for XPS is shallower than EDS

Direct Thermal Reduction: Material Characterization

LHS-1 – EDS

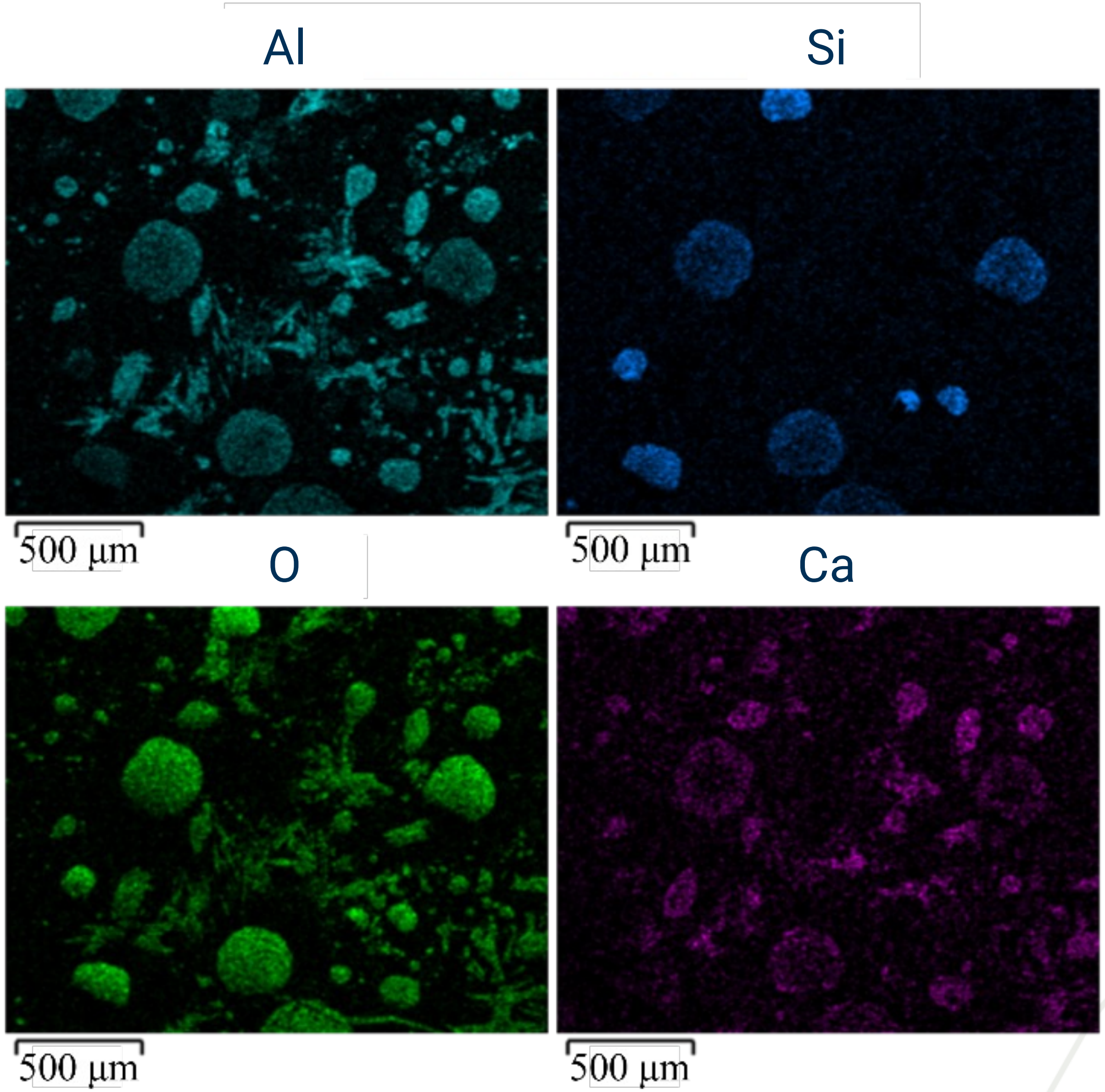
- Depositions were evenly distributed on Cu foils
- Na, Fe, and K evolved during both experiments suggesting separation possibility at reasonable temperatures
- Si and Mg evolved continuously, and Al and Ca remained in sample upon experimentation evidencing requirement of elevated temperatures



Direct Thermal Reduction: Material Characterization

LHS-1 – EDS

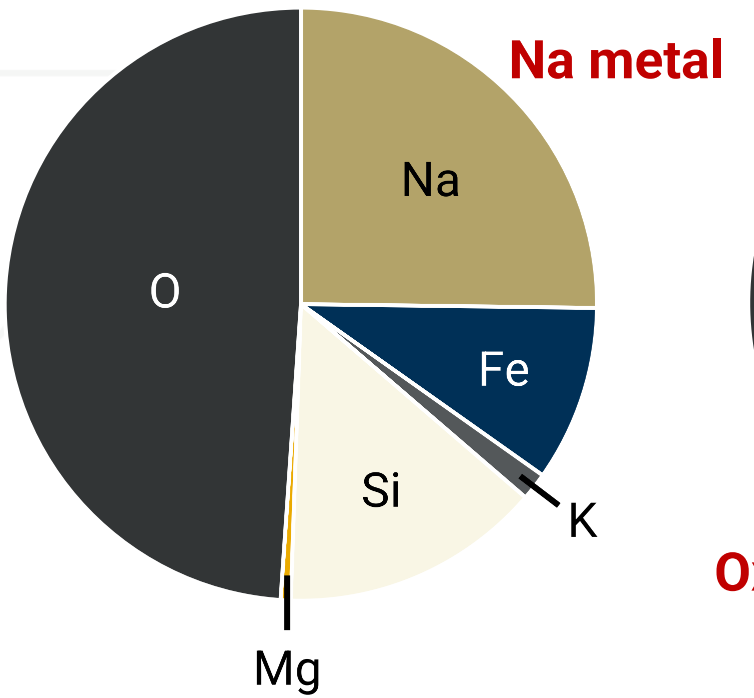
- Sample segregation observed on remaining sample
- Significant amounts of sample containing Al and Ca without Si, suggesting potential Al production
- The elements detected are likely oxides, indicated by the O scan



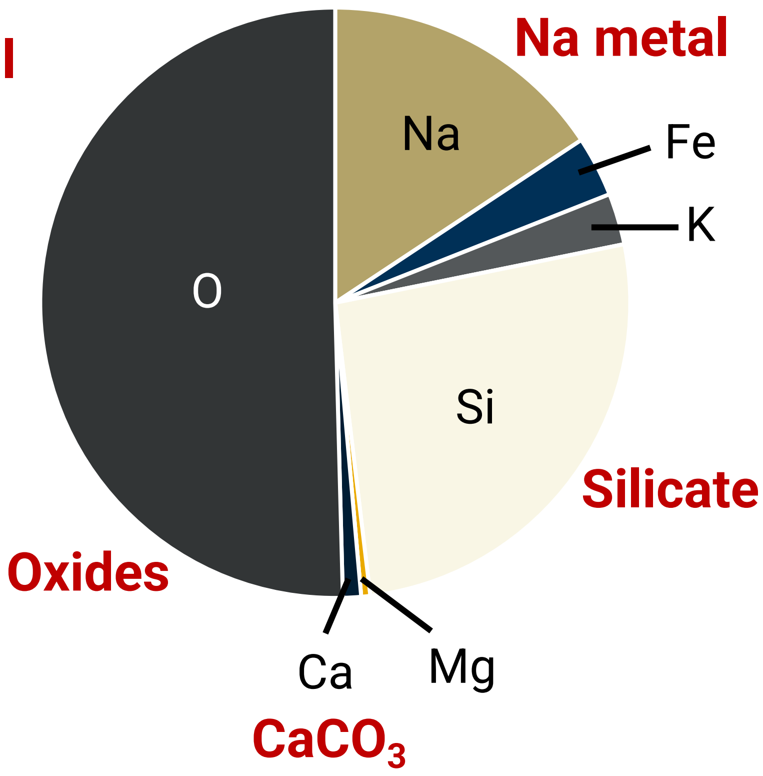
Direct Thermal Reduction: Material Characterization

LHS-1 – XPS

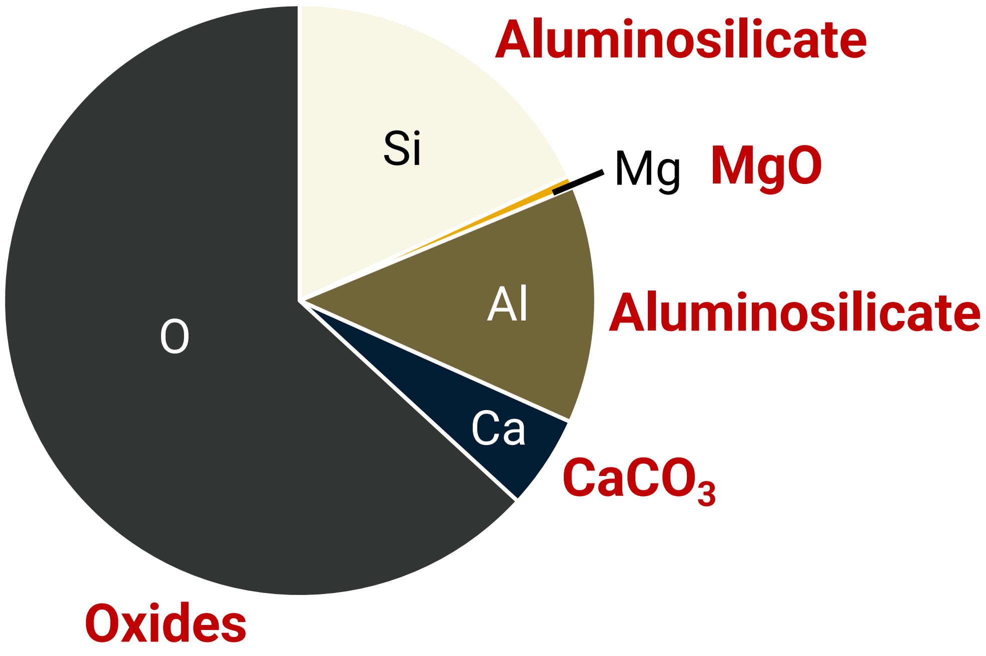
P = 835.9 W



P = 985.5 W



Remaining sample

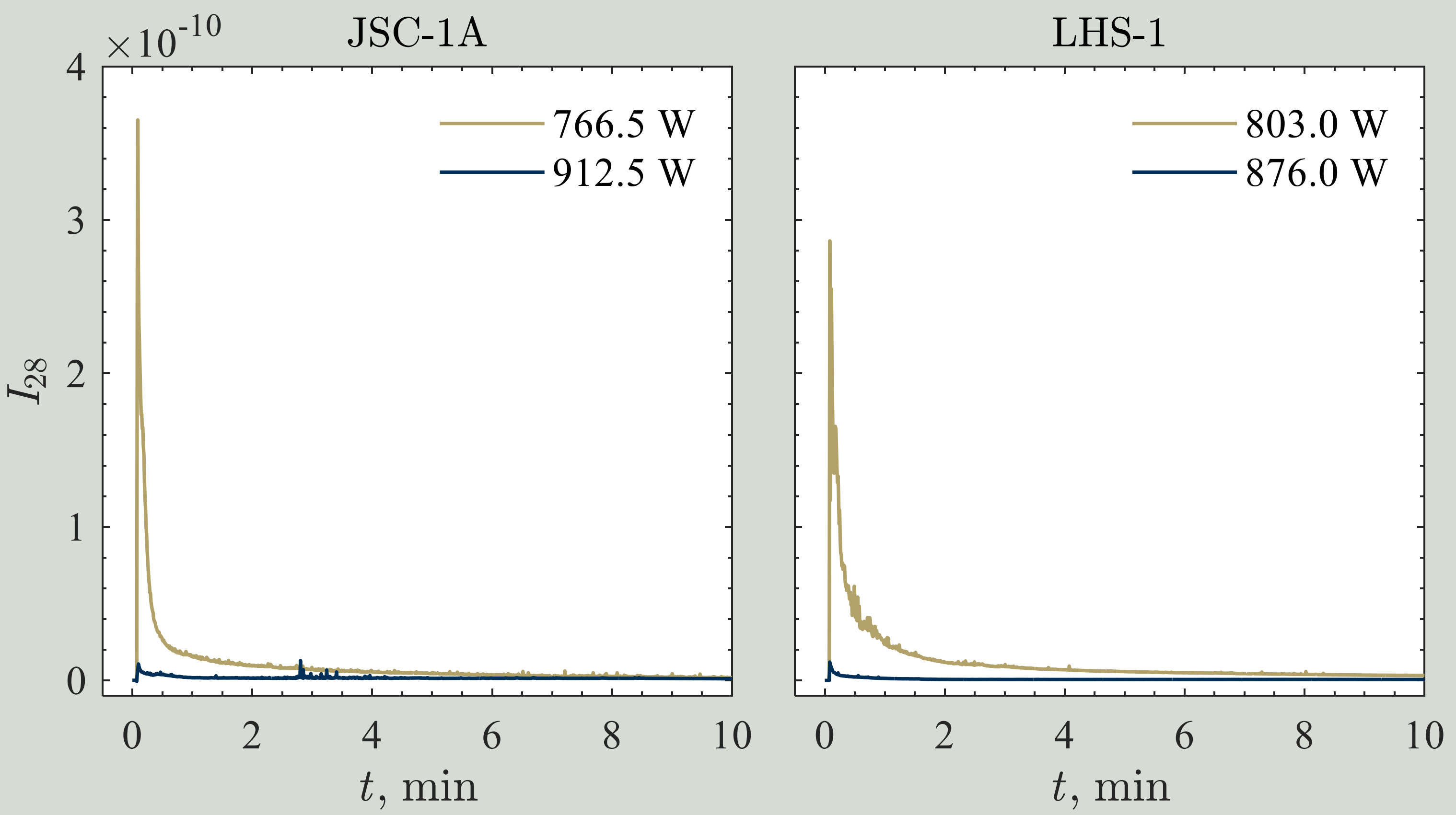


- Depth of analysis for XPS is shallower than EDS

Carbothermal Reduction

- CO(g) evolution from sample during experiments was evidenced by the QMS at 28 amu
- A prominent peak at t ≤ 0.5 min was observed with no significant changes in I_{28} for t > 10 min, indicative of rapid thermal reductions
- Smaller amounts of CO(g) evolved during the second experiments because the reacted sample from first experiments were used causing smaller sample size

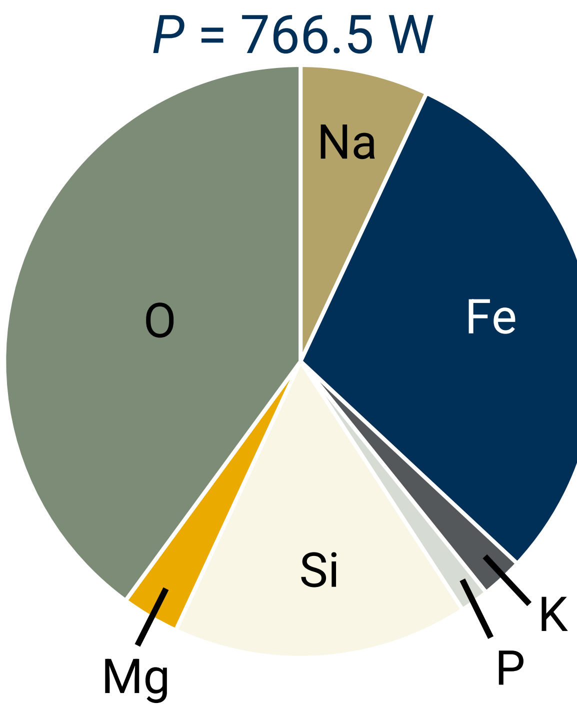
Ionic current of amu 28 as measured by QMS

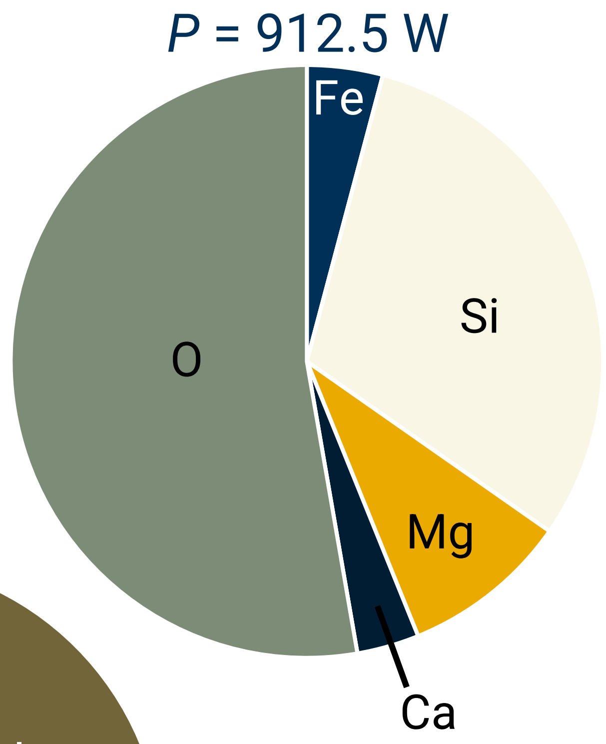


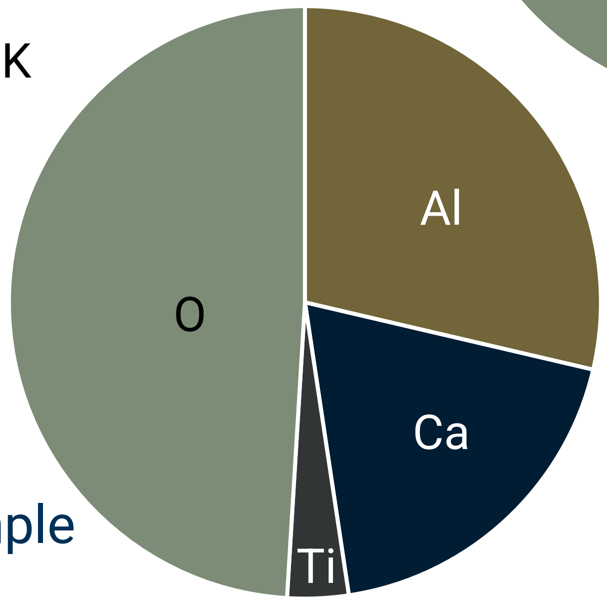
Carbothermal Reduction: Material Characterization

JSC-1A + C – EDS

- Depositions were evenly distributed on all foils
- Na, Fe, K, P, Si and Mg evolution during experiments and absence in remaining sample evidenced complete vaporization
- Al, Ca, and Ti remained in sample upon experimentation suggesting separation and production possibility of these elements



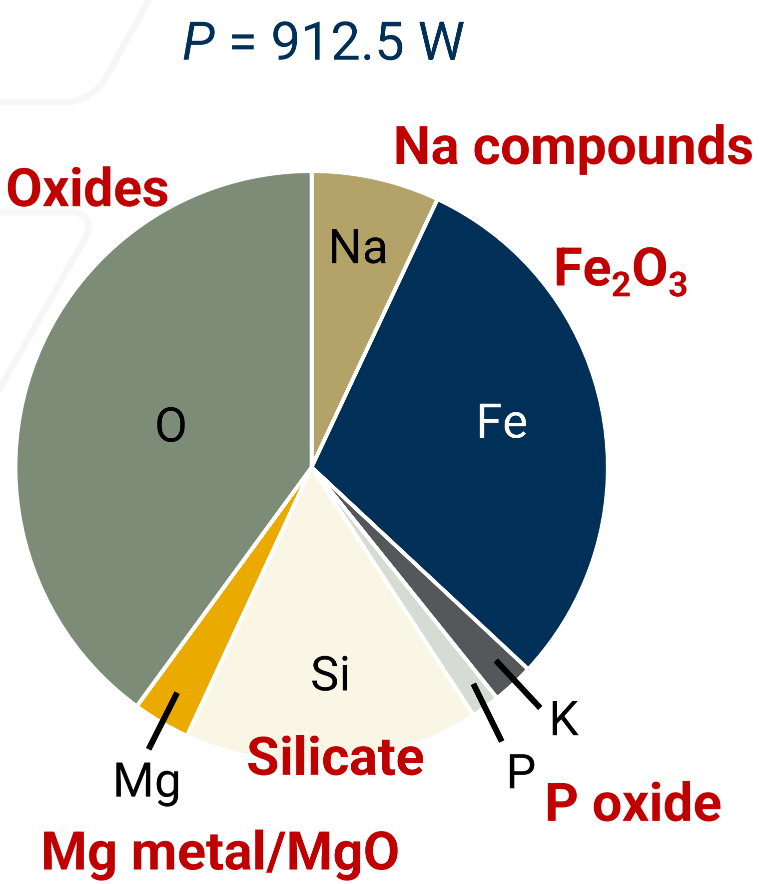


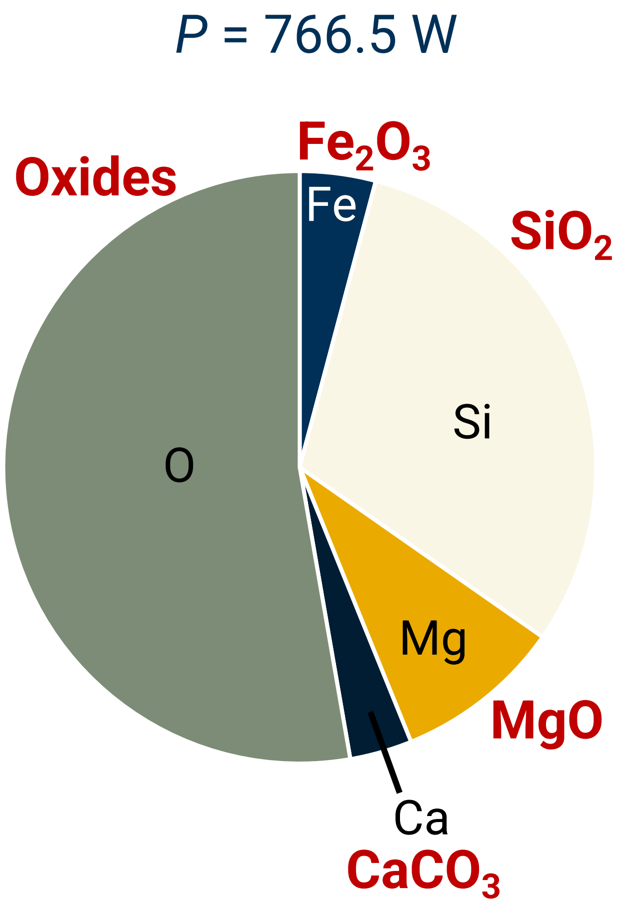


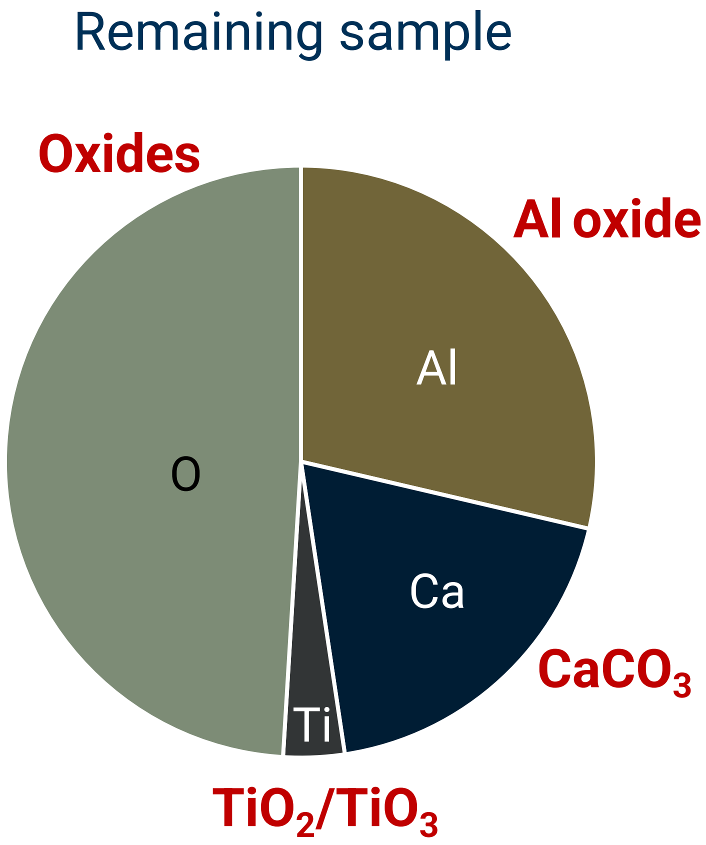
Remaining sample

Carbothermal Reduction: Material Characterization

JSC-1A + C – XPS





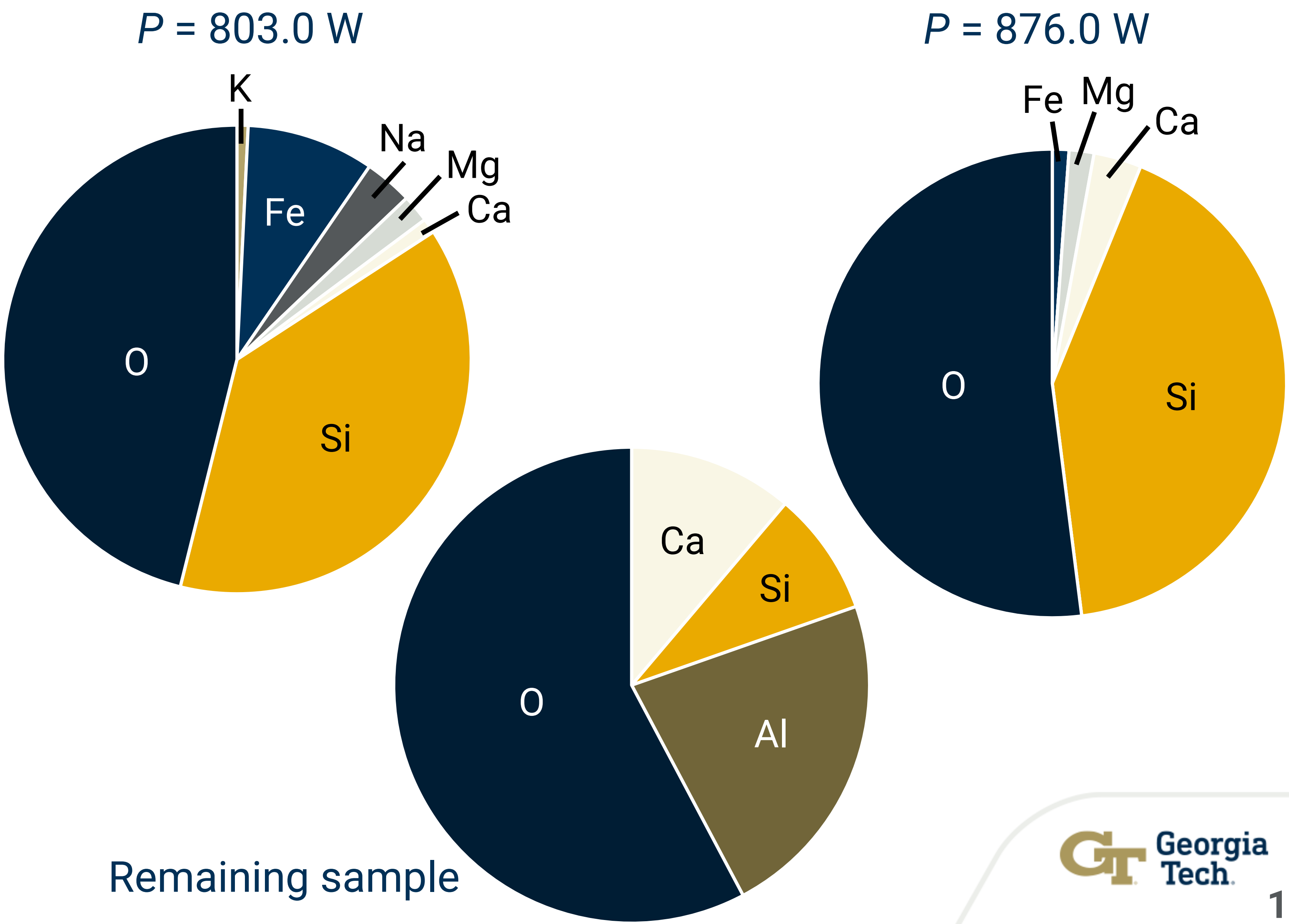


- Depth of analysis for XPS is shallower than EDS

Carbothermal Reduction: Material Characterization

LHS-1 + C – EDS

- Depositions were evenly distributed on Cu foils
- Na, Fe, and K evolved during the first experiment suggesting separation possibility at reasonable temperatures and complete vaporization
- Si, Mg, and Ca evolved continuously, and Al, Si, and Ca remained in sample



Carbothermal Reduction: Material Characterization

LHS-1 + C – EDS

- Sample segregation observed on remaining sample
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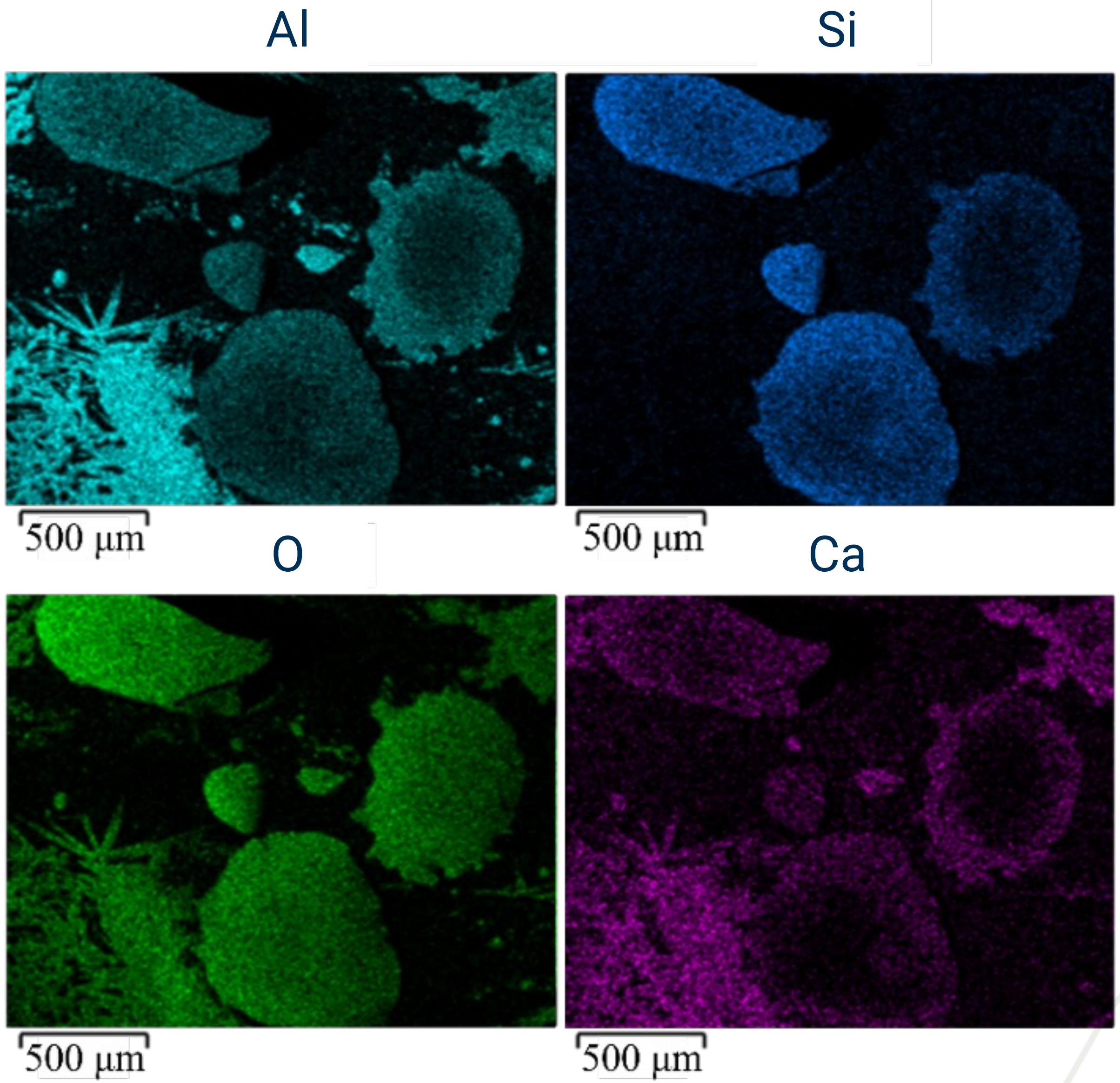
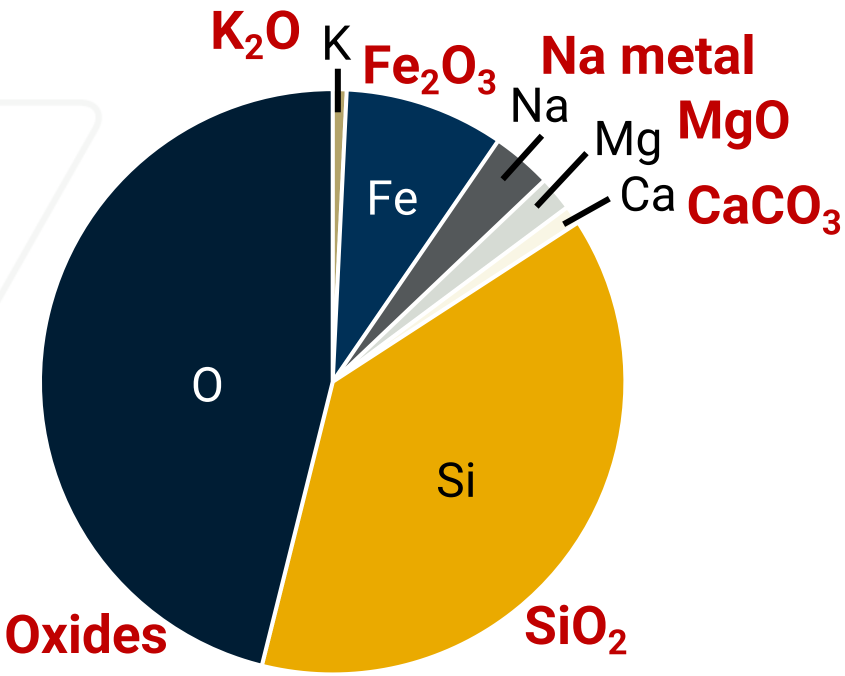
- The elements detected are likely oxides, indicated by the O scan



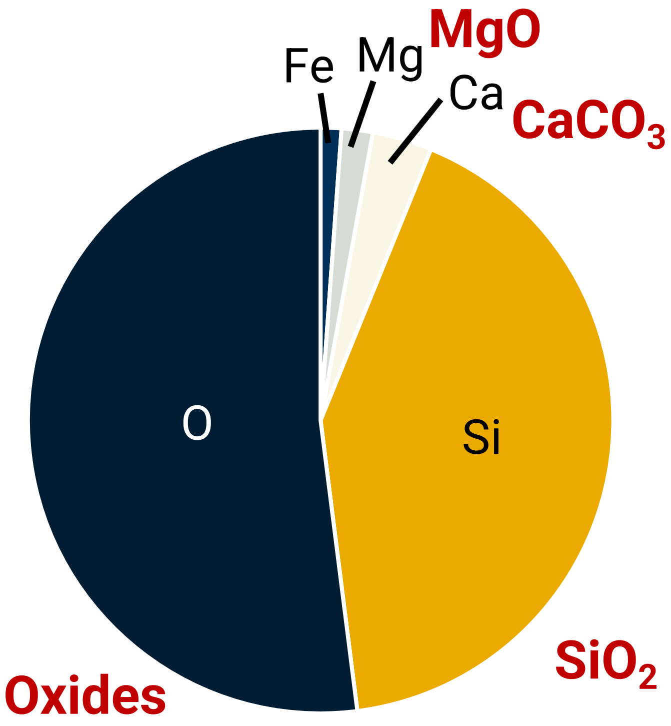
Direct Thermal Reduction: Material Characterization

LHS-1 + C – XPS

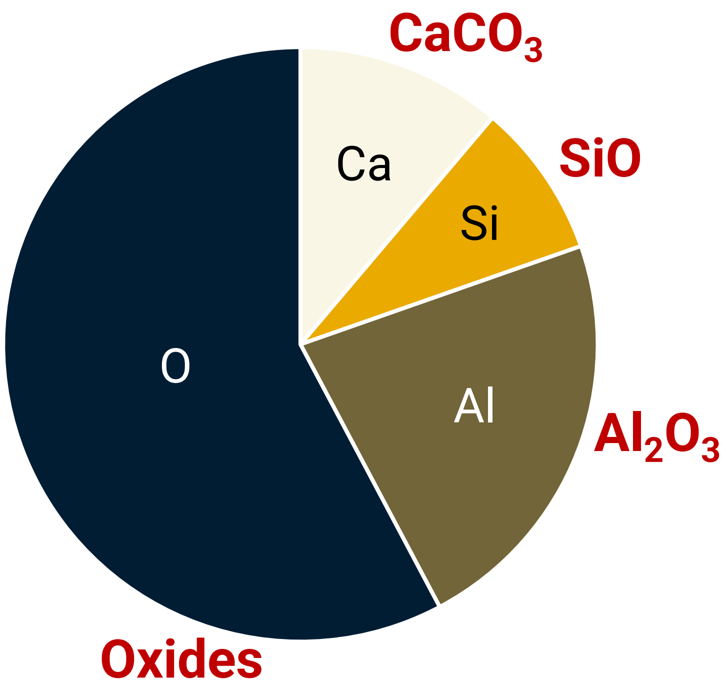
P = 803.0 W



P = 876.0 W



Remaining sample



- Depth of analysis for XPS is shallower than EDS

Summary and Conclusions

- Experimental analyses on thermal and carbothermal reduction of lunar regolith simulants for O_2 and metal/metalloid production in ultra-high vacuum conditions
- Oxygen production was evident in the form of O_2(g) for thermal reduction and CO(g) for carbothermal reduction
- Characterization of vapor collecting foils upon experimentation showed that Na, Fe, K, and P are extractable at relatively lower temperatures whereas Si, Mg, and Ca require elevated temperatures
- Characterization of remaining samples upon experimentation showed the possibility of Al separation and production, especially from highlands
- Larger quantities and more compounds vaporized via carbothermal than thermal reduction because reaction onset temperatures were reduced, evidencing the benefit of adding a reductant

Acknowledgements

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Research Team:

- Solar Fuels and Technology Laboratory (Solar FTL) at Georgia Tech
- Electron Proton Induced Chemistry on Surfaces (EPICS) laboratory at Georgia Tech

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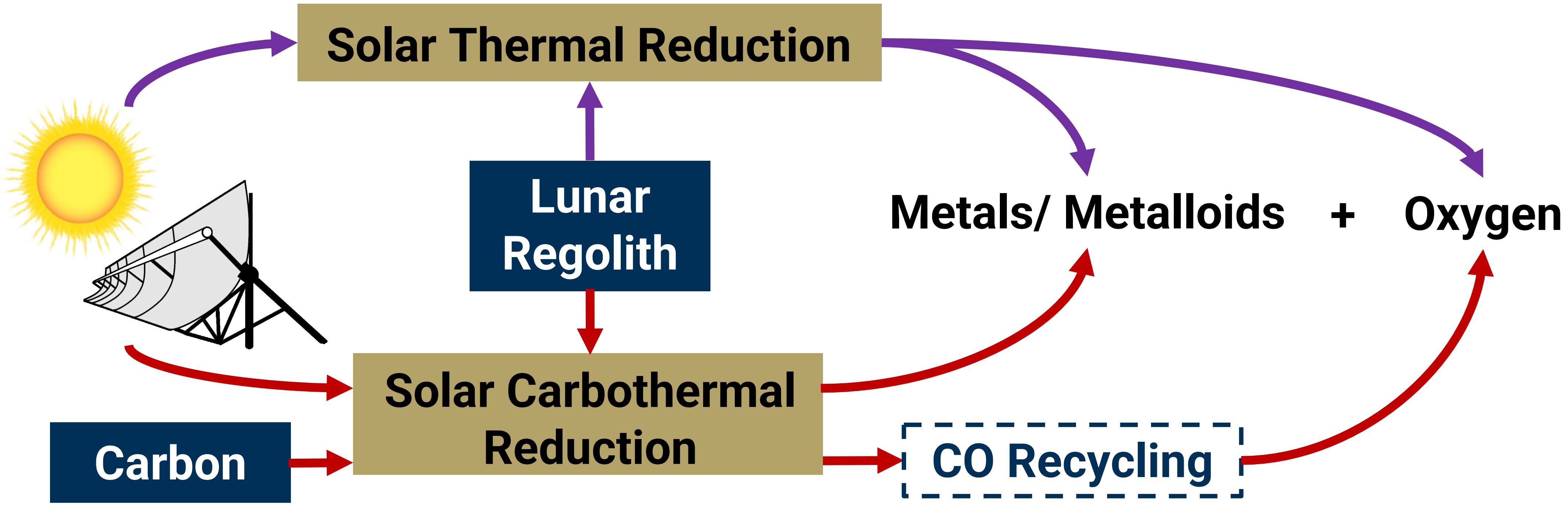
Thank you for listening!



Questions?

Oxide Reaction Equations

Direct Thermal

Oxide	Reaction
SiO_2	$SiO_2 \rightarrow Si + O_2(g)$
Al_2O_3	$2Al_2O_3 \rightarrow 4Al + 3O_2(g)$
FeO	$2FeO \rightarrow 2Fe + O_2(g)$
CaO	$2CaO \rightarrow 2Ca + O_2(g)$
MgO	$2MgO \rightarrow 2Mg + O_2(g)$
Na_2O	$2Na_2O \rightarrow 4Na + O_2(g)$
TiO_2	$TiO_2 \rightarrow Ti + 2O_2(g)$
K_2O	$2K_2O \rightarrow 4K + O_2(g)$
P_2O_5	$2P_2O_5 \rightarrow 4P + 5O_2(g)$
MnO	$2MnO \rightarrow 2Mn + O_2(g)$
Cr_2O_3	$2Cr_2O_3 \rightarrow 4Cr + 3O_2(g)$

Carbothermal

Oxide	Reaction
SiO_2	$SiO_2 + 2C \rightarrow Si + 2CO(g)$
Al_2O_3	$Al_2O_3 + 3C \rightarrow 2Al + 3CO(g)$
Fe_2O_3	$Fe_2O_3 + 3C \rightarrow 2Fe + 3CO(g)$
CaO	$CaO + C \rightarrow Ca + CO(g)$
MgO	$MgO + C \rightarrow Mg + CO(g)$
Na_2O	$Na_2O + C \rightarrow 2Na + CO(g)$
TiO_2	$TiO_2 + 2C \rightarrow Ti + 2CO(g)$
K_2O	$K_2O + C \rightarrow 2K + CO(g)$
P_2O_5	$P_2O_5 + 5C \rightarrow 2P + 5CO(g)$
MnO	$MnO + C \rightarrow Mn + CO(g)$
Cr_2O_3	$Cr_2O_3 + 3C \rightarrow 2Cr + 3CO(g)$