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Life Support Catalyst Regeneration Using Ionic Liquids and In Situ Resources

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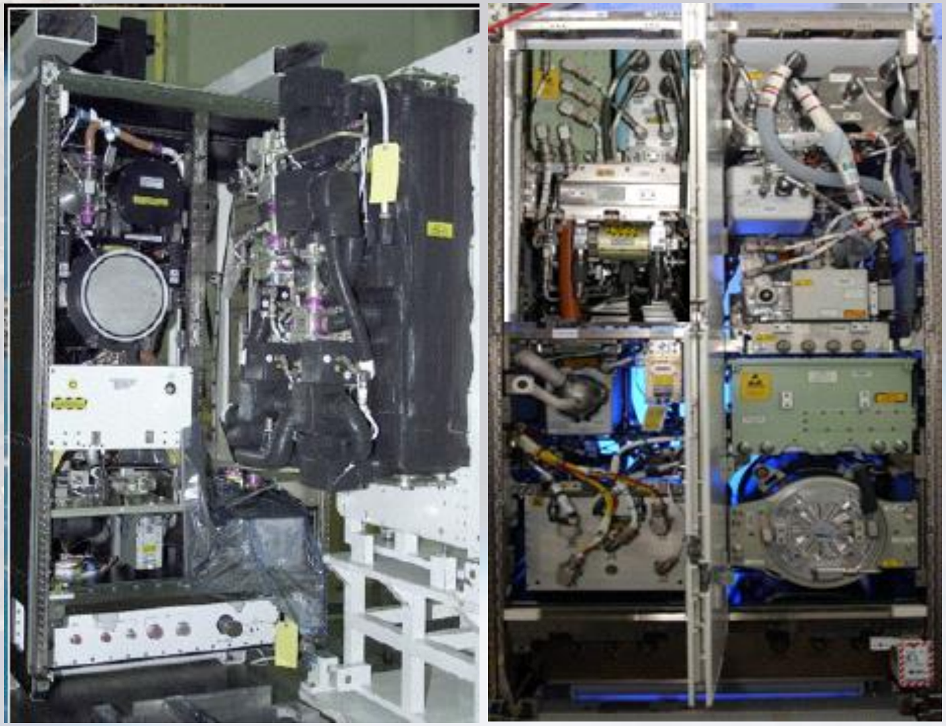
Agenda

- Background
- Project Goals
- Methods
- Results
- System Concept for Surface Mission
- Key Challenges and Future Work

Background

- Environmental Control and Life Support (ECLSS) Functions
  - Temperature and Humidity Control
  - Trace Contaminant Control
  - Oxygen Generation
  - Carbon Dioxide Removal
  - Oxygen Recovery
- Oxygen Recovery
  - SOA ~50% O_2 from metabolic CO_2
  - Long duration missions targeting >90%



ISS Atmosphere Revitalization Rack (left) and Oxygen Generation System Rack (right)

Background

- Achieving up to 100% O_2 recovery

$$CO_2 \leftrightarrow O_2 + C(s)$$

- One pathway is $CO_2 \rightarrow CO \rightarrow C(s)$
  - Making CO:

$$CO_2 + H_2 \leftrightarrow H_2O + CO$$

$$2CO_2 \leftrightarrow O_2 + 2CO$$

Various technology options available to do this

  - Making solid carbon:

$$CO + H_2 \leftrightarrow H_2O + C(s)$$

$$2CO \leftrightarrow CO_2 + C(s)$$

Targeted Development

Background

- Solid Carbon Production
  - Carbon formation occurs over Fe, Ni, Co, and alloy catalysts at 400-650°C
  - Fast reaction rates at higher temperatures (kinetically favorable)
  - More carbon formation at lower temperatures (thermodynamically favorable)
  - Carbon eventually fouls the catalyst

HIGHLY UNFAVORABLE FOR LONG DURATION MISSIONS DUE TO CATALYST RESUPPLY MASS

Background

- Ionic Liquids
  - Organic salts that are liquid at temperatures $\leq$ 100°C (as defined here)
  - Virtually no vapor pressure
  - Low flammability
  - Can be extensively chemically modified to be task-specific
  - Stable under extreme conditions (vacuum and cold temperatures)
  - Regenerable
  - Work at temperatures below 200°C.
- Previous work by Karr *et. al.* demonstrated IL extraction of Ni and Fe from an asteroid and plating on a new surface
- Hypotheses:
  1. IL may be used to EXTRACT Fe from Martian or Lunar regolith to resupply exhausted catalyst (eliminates Earth resupply)
  2. IL may be used to PRODUCE an Fe catalyst (*in situ* production capability)
  3. IL may be used to REGENERATE an Fe catalyst (reduces catalyst resupply required)

Background

EXTRACT

IL + H^+

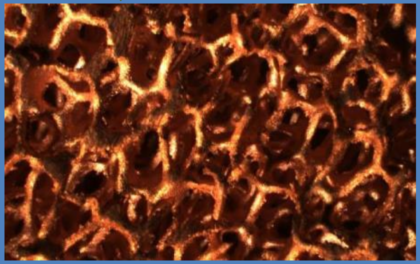
IL + Fe

H_2

PRODUCE

IL + Fe

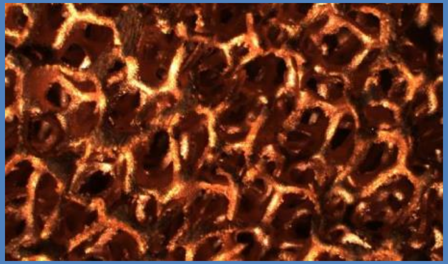
H_2



IL + H^+

REGENERATE

IL + H^+



IL + Fe

C(s)

H_2

Project Goals

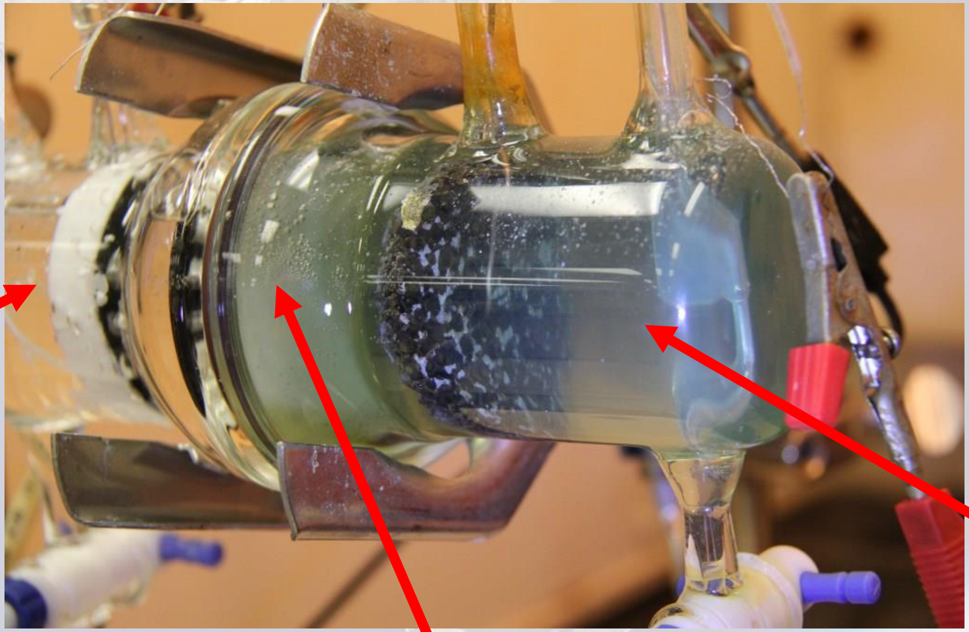
1. To demonstrate catalytic activity of iron (Fe) on a copper (Cu) support, electroplated using traditional methods
2. To demonstrate extraction of Fe from Fe-Carbon (C) product using IL
3. To demonstrate plating of Fe on Cu support, plated using IL
4. To demonstrate catalytic activity of Fe on Cu support, plated using IL
5. To demonstrate catalytic activity of IL-plated and regenerated Fe over several cycles

Methods

- Fe Plating Solutions
  - FeSO4 (traditional plating solution)
  - Aqueous solution of the acid salt ammonium hydrogen sulfate $[NH_4][HSO_4]$
  - $[bmpyrr][HSO_4]$
  - $[emim][HSO_4]$
  - $[mpyrr][HSO_4]$
    - Safety advantages (low vapor pressure, low flammability, etc.)
    - Sufficiently acidic to dissolve iron metal to Fe+2 (the acidity comes from the HSO4- anion of the ILs)
    - Similar ILs shown to be good at solvating carbon nanotubes, thus possibly aiding in removal of the carbon deposits from the Cu pucks
    - Electrochemical windows wider than water – will not break down during Fe plating

Methods

- Plating
  - 4 hrs for plating each side of Cu substrate (puck)



Anode containing Pt cloth

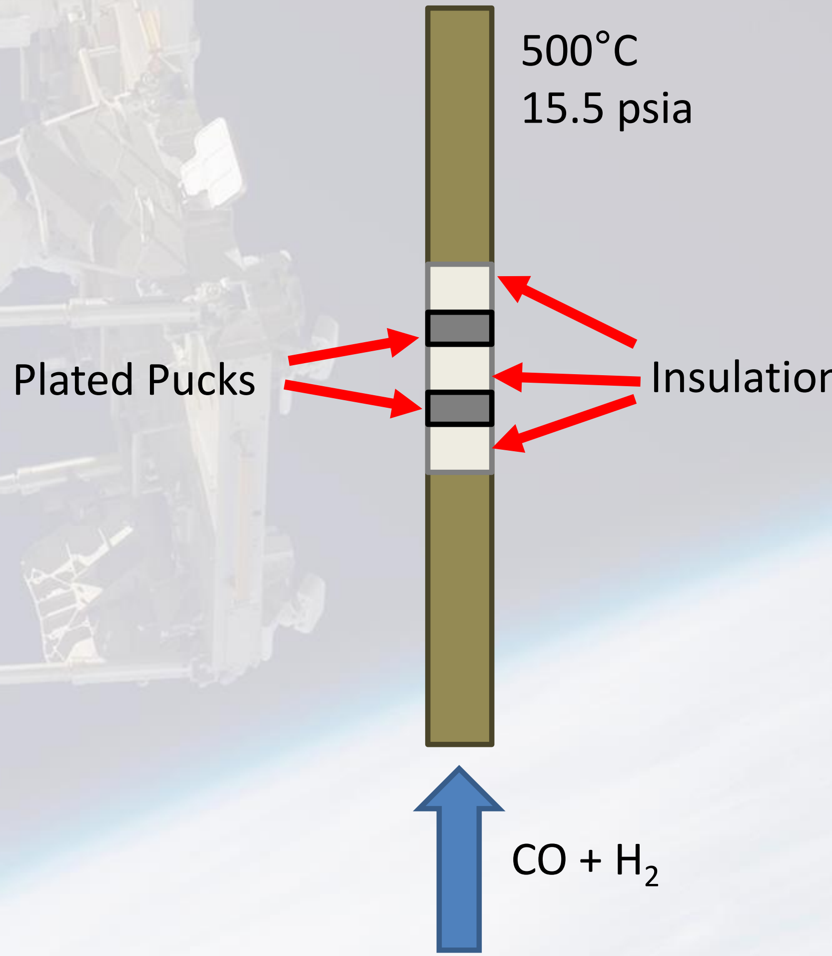
Quaternary ammonium-functionalized polystyrene anion exchange membrane

Cathode containing ERG Cu foam substrate (puck)

Methods

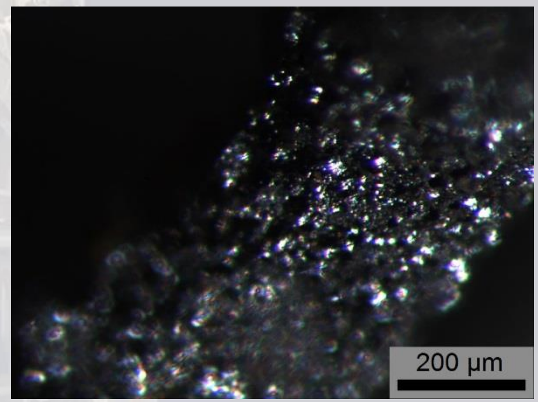
- Carbon Formation
  - CO_2 Reduction Catalyst Test Stand (COR-CaTS)

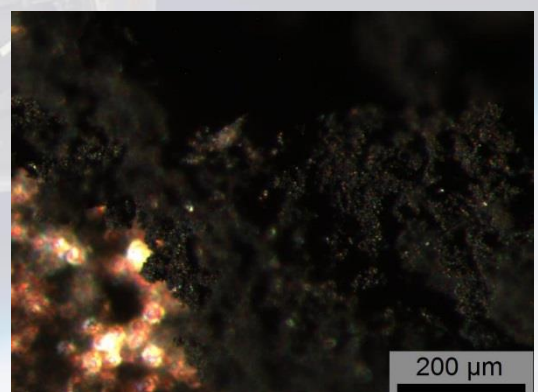
COR-CaTS



Results

- Goal 1: Demonstrate catalytic activity of traditionally plated iron Cu support

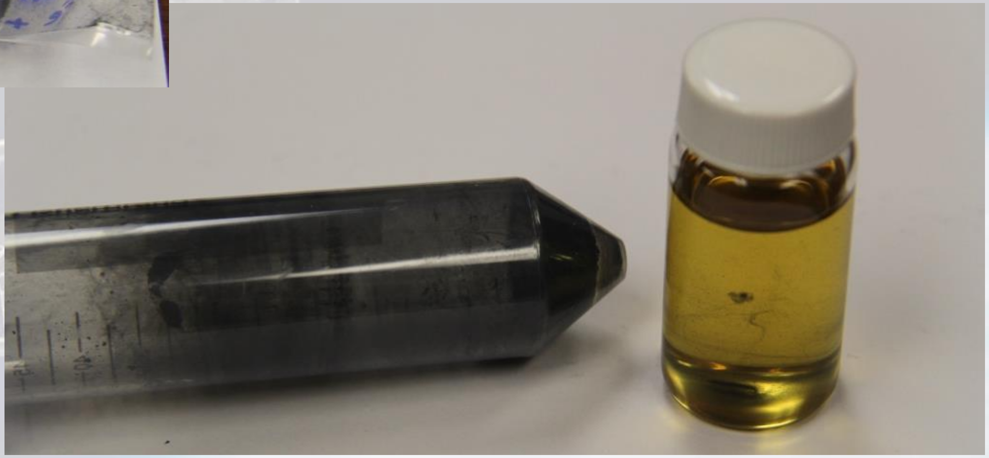




Results

- Goal 2: Demonstrate extraction of Fe from Fe-Carbon (C) product using IL

Fe wool coated in carbon during previous Bosch catalyst development testing.



Carbon residue following IL extraction of Fe (left) and IL containing Fe with small quantities of residual carbon (right).

Results

- Goal 3: Demonstrate plating of Fe on Cu support, plated using IL

Raw copper substrate

Fe-coated copper from [bmpyrr][SO_4]

Fe-coated copper from [emim][SO_4]

Fe-coated copper from [mpyrr][SO_4]

Results

- Goal 4: Demonstrate catalytic activity of Fe on Cu support, plated using IL

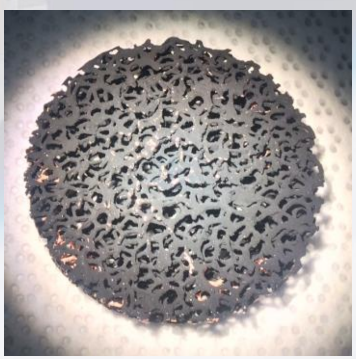
Freshly plated pucks

Fe-coated copper from [bmpyrr][SO_4]

Fe-coated copper from [emim][SO_4]

Fe-coated copper from [mpyrr][SO_4]

Pucks after carbon formation

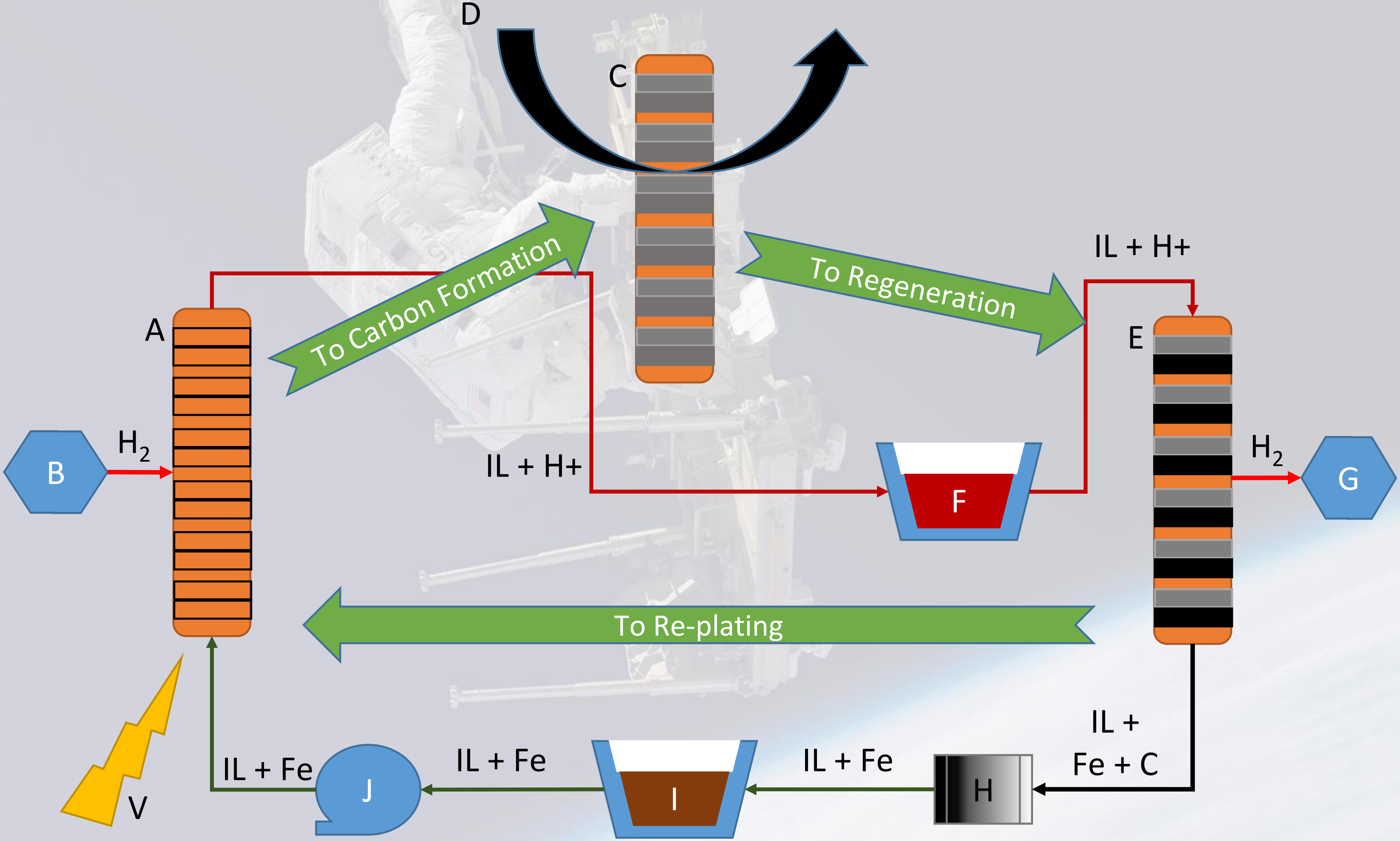


Results

- Goal 5: Demonstrate catalytic activity of IL-plated and regenerated Fe over several cycles

Include new regen data from Steve.

Surface System Concept



Key Challenges & Future Work

1. Even distribution of Fe plated on the Cu substrate with multiple electrodes
   – Pulsed power supply
2. Thermal variation between carbon formation and electroplating/regeneration
   – Carbon formation = 450°C, electroplating and regeneration at room temperature
   – Material selection for high temp and thermal cycling
   – Or separate chambers for each process, which results in more complexity and more challenges due to leak potential with H_2, CH_4, and CO gases.
3. Carbon handling
   – 1.1 kg of carbon/day for a crew of 4
   – 5 year mission = 2000 kg of carbon
4. IL Cleaning following regeneration
   – Sufficient cleaning necessary for replating
5. Sizing
   – Large enough to handle volume of carbon
   – Small enough to achieve reasonable power and cooling requirements for cyclic operation

Questions?

Backup

- [bmpyrr][HSO_4] = N-butyl-N-methylpyrrolidinium hydrogen sulfate
- [emim][HSO_4] = 1-ethyl-3-methylimidazolium hydrogen sulfate
- [mpyrr][HSO_4] = N-methylpyrrolidinium hydrogen sulfate