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Pulsed Electrodeposition of Carbon Dioxide Reduction Electrocatalysts for Space Applications

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Terrestrial Motivation

- **CO₂ Conversion**

- Environmental concerns
- Abundant carbon feedstock for value-added products
 - CO, formate, hydrocarbons
- Point sources (e.g., power generation)
 - Co-location of capture & conversion
- Direct conversion of gas-phase CO₂
 - Gas-diffusion electrode (GDE) electrolyzer

NASA Relevance

Pulse/Pulse-Reverse Electrodeposition

Pulsed Waveforms – Enhanced Process Control

Electrochemical Machining, Polishing, Deburring, Through-Mask Etching

Anodic Pulse “Tuned” to:

- Control current distribution

→ Eliminates need for viscous, low water content electrolytes

Cathodic Pulse “Tuned” to:

- Reduce oxide/depassivate surface

→ Eliminate need for HF

Electrodeposition/Plating, Electrocatalysis

Cathodic Pulse “Tuned” to:

- Enhance mass transfer
- Control current distribution

→ Simplify chemistry

Anodic Pulse “Tuned” to:

- Remove H₂ effects

→ Acidify interface

E.J. Taylor “Adventures in Pulse/Pulse Reverse Electrolytic Processes: Explorations and Applications in Surface Finishing” J. Appl. Sur. Fin. 3(4) 178-89 (2008).

Prior Work: Lead-Free Tin

Rotating Disk Plating Tool

Sn Deposits on Cu Substrates — 1000× Magnification

U.S. Patent 8,603,315 Granted

Prior Work: Copper Electrodeposition for PCBs

Standard DC Recipe Pulsed Waveform Recipe

CuSO_4
 H_2SO_4
 "Accelerator"
 "Suppressor"
 "Leveler"
 Other Additives

CuSO_4
 H_2SO_4
 Trace Cl^-
 Trace PEG

Plante, et al. "Quantization and Characterization of Copper Plating Bath Additives by Liquid Chromatography with Charged Aerosol Detection." Thermo Scientific. http://www.dionex.com/en-us/webdocs/113469-Pittcon12_1820-6_MPlante_PN70008_PlatingBaths.pdf

*Z-Interconnects Cu-Plated by Pulse-Reverse
 FARADAYIC® ElectroDeposition*

Prior Work: NiMo Functionally-Graded Coating

Functionally graded NiMo coatings for improved corrosion resistance in molten-fluoride-salt nuclear reactors (MSRs)

FARADAYIC[®] ElectroCatalyzation

Gas Diffusion Electrode (GDE) Electrocatalysts

FARADAYIC[®] GDE ElectroCatalyzation

TEM of Pt on Carbon

Performance Comparison

FARADAYIC® GDE ElectroCatalyzation

Electronic + Ionic Contact

Electrochimica Acta 39(11-12): 1833 (1994).

CO₂ GDE Catalyst Product Selectivity

Shifts in product distribution over copper-alloy electrocatalysts vs pure Cu as reviewed by Nitopi et al.
Chemical Reviews 119: 7672 (2019).

Electrochimica Acta 39(11-12): 1833 (1994).

GDE Catalysts – Sn

GDE Catalysts – Cu

Electrocatalyst Testing

 

Flow Cell Apparatus

Sn GDE – W-Cell Electrocatalsysis

(Half-cell potentials vs SHE)

Li and Oloman. *J Appl Electrochem* 35: 955, 2005.

Agarwal et al. *ChemSusChem* 4: 1301, 2011.

Kopljar et al. *J Appl Electrochem* 44: 1107, 2014.

Sen, S. et al. "Pulsed Electrodeposition of Tin Electrocatalysts onto Gas Diffusion Layers for Carbon Dioxide Reduction to Formate." *MRS Adv* 2(8): 451 (2017).

Cu GDE – Flow Cell Electrocatalysis

Sen, S. et al. "Pulse Plating of Copper onto Gas Diffusion Layers for the Electoreduction of Carbon Dioxide." *MRS Adv* 3(23): 1277 (2018).

Summary

- Wide variety of active GDE catalyst materials can be deposited via pulsed FARADAYIC[®] ElectroCatalyzation
 - Readily scalable process
- High-activity CO₂ conversion catalysts
 - Demonstrated in industrially-relevant, scalable form factor
 - Performance competitive with or superior to literature
 - Total activities (mA/cm²)
 - Formate/ethylene selectivity
 - Durability appears promising; further testing is pending
- Reactor/GDL development in progress
 - Goal: 100+ to 1000+ hours of steady operation
 - Goal: Optimization of Sn ElectroCatalyzation parameters

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Thank You!