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Pulsed Electrodeposition of Gas-Diffusion Electrocatalysts for CO₂ Reduction

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Motivation

- **CO₂ Conversion**
 - Environmental concerns
 - Abundant carbon feedstock
 - CO, formate, hydrocarbons
 - Power generation → Point sources
 - Co-location of capture & conversion

CO₂ Conversion – Form Factor Selection

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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 H_2 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

CuSO_4
 H_2SO_4
“Accelerator”
“Suppressor”
“Leveler”
Other Additives

Pulsed Waveform Recipe

CuSO_4
 H_2SO_4
Trace Cl^-
Trace PEG

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

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

FARADAY TECHNOLOGY, INC.

FARADAYIC[®] ElectroCatalyzation

Gas Diffusion Electrode (GDE) Electrocatalysts

FARADAYIC[®] GDE ElectroCatalyzation

TEM of Pt on Carbon

Performance Comparison

FARADAYIC® GDE ElectroCatalyzation

Electronic + Ionic Contact

CO₂ Electrocatalyst Selection

Product distribution over single-metal electrocatalysts as determined by Hori et al.

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

e											B	C	F
g											Al	Si	P
a	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As
r	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb
a	*	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi

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GDE Catalysts – Sn

GDE Catalysts – Cu

Electrocatalyst Testing

W-Cell Apparatus

Flow Cell Apparatus

Sn GDE – W-Cell Electro catalysis

(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).

Electroreactor Development

Reactor/GDL Development

- Industrial durability
 - Must maintain performance for 1000s of hours
- Initial durability challenge:
Excessive electrolyte penetration through GDE
- Anticipated factors:
 - GDL/GDE porosity, hydrophobicity
 - Reactor design/configuration
 - gas-liquid pressure balance

Gas Diffusion Layer Fabrication Parameters

Breakthrough Pressure Apparatus

- Custom-fabricated cell to detect onset of GDL liquid breakthrough
- Breakthrough pressure = proxy for electrochemical performance?
- Continuous monitoring by video
- Pressure ramp controlled by upstream pump drive rate

Preliminary Breakthrough Pressure Data

Preliminary Breakthrough Pressure Data

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

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Thank You!