

Electrodeposited Mn-Co Alloy Coating for SOFC Interconnects

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> 13th Annual SECA Workshop July 25, 2012

Faraday Technology, Inc.

- Faraday Technology specializes in electrochemical engineering
 - www.faradaytechnology.com
- Faraday is a wholly-owned subsidiary of Physical Sciences, Inc. (Boston, MA)
 - www.psicorp.com

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- Collectively, the company staffs ~185 employees - ~100 with PhDs
- Annual revenue of ~ \$50M



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Platform Technology: Pulse/Pulse Reverse Processing



Core Competency: Design and Engineer of Novel Electrochemical Hardware



Either may be applied independently to improve current industrial practices or may be combined for a total manufacturing solution

- Electronics
- Edge and Surface Finishing
- Engineered Coatings
- Battery and Fuel Cell Power
- Environmental Systems
- Corrosion and Monitoring Services

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- Enables uniform processing
- Applicable for additive or subtractive electrochemical processes
- Uniform processing is achieved over entire substrate, improving end product reliability

Achievements

- Continued optimization of FARADAYICSM Electrodeposition Process parameters in order to optimize coating thickness, coating composition and coating adhesion
- Improved coating uniformity across T441 planar interconnects at the 100 cm² scale
- Demonstrated coating process for 25 cm² 430 stainless steel interconnect containing gas flow fields
- Continued refinement of economic analysis to assess economic viability of FARADAYICSM Electrodeposition Process for high volume batch manufacturing

FARADAYICSM Processing

Conventional (DC) Electrodeposition

FARADAYICSM Process



- Fast deposition rates
- Simple deposition equipment
- Non-line-of-sight deposition
- Industrially scalable



- Improved electric field control
 - Enhanced control of coating thickness uniformity
 - Enhanced control of alloy composition
- Improved coating of "hidden surfaces"

Coating Process

- Surface pretreatment to remove oxide and enhance coating adhesion
- Electrodeposition to coat interconnects with Mn-Co alloy
 - Pulse and pulse reverse electric fields to control deposit properties
- Elevated thermal treatment to convert alloy to spinel

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Phase I Hull Cell Experiments

Enables investigation into the effect of various parameters on deposit properties during a single experiment

- Current density
- Temperature
- Electrolyte composition
- Additives







Phase I Hull Cell Experiments

Electrolyte with NaC₆H₁₁O₇



Electrolyte without NaC₆H₁₁O₇



- Electrolyte without NaC₆H₁₁O₇ was selected for Phase I work on 5 cm x 5 cm T441 planar substrates because at reasonable current densities and metal ion concentrations results suggested
 - Potential for higher Mn content in coating
 - Less microcracking
 - Higher current efficiency
 - Faster coating deposition rates

Phase I Cr Diffusion and Coating Porosity



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- Cross-sections of samples that underwent a soak treatment at 800 C for 500 hrs.
 - Coating thickness was as deposited
 - Indicates that the 3 micron layer has low Cr diffusion and the 10 micron coating has negligible Cr diffusion into coating
 - 3 micron coating appears more porous than 7 and 10 micron film

Phase I Coating Crystal Structure



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Phase I Effect of Thickness and Composition on Performance

The ASR is $\leq 60 \text{ m}\Omega \text{ cm}^2$ in most cases regardless of compositions and thickness after 500 hrs. at 800 C



ASR at 800 C

$m\Omega \text{ cm}^2$	100 hr	500 hr	
3 μm 40% Co	35	49	
7 μm 40% Co	62	32	
10 μm 40% Co	22	36	
3 μm 85% Co	31	20	
7 μm 85% Co	59	54	
10 μm 85% Co	37	22	
3 μm 57% Co	-	26	
7 μm 57% Co	_	12	
10 µm 57% Co	-	12	

Phase III Program Management Plan

	Milestones						
Fiscal Year	Title	Planned Completion	Percent Complete				
2011	1. Design/modification of 10" x 10" electrodeposition cell	May 2011	100%				
2012	2. Long-term high temperature, thermal evaluation	August 2012	70%				
2012	3. Process development for 4"x4" planar interconnects	May 2012	100%				
2012	4. Process development for 4"x4" pattern interconnects	June 2012	10%				
2012	5. Long-term on-cell performance evaluation	August 2012	10%				
2012	6. Qualification/demonstration of IC in single cell test rig	September 2012	0%				

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Pilot Scale Electrodeposition Equipment





Based upon Faraday's electrochemical cell design that facilitates uniform flow across the surface of a flat substrate (US patent #7,553,401)



Pilot Scale Experiments

- After several tests, issues were noticed with coatings
 - Non-uniform current density on front and back of sample during plating
 - Poor chemical composition control
 - Coating thickness non-uniformity
 - Poor coating adhesion
- Anodes removed from system
 - Mn fouling

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- High surface resistivity
 - In the megaohm range after only a few tests
- Can be removed with 30% (v/v) sulfuric acid



NaC₆H₁₁O₇ Electrolyte Revisited

- Addition of $NaC_6H_{11}O_7$ to electrolyte
 - Observed benefits
 - Boric acid dissolves completely
 - Complexing metal ions prevents hydroxide formation
 - Improved buffer capacity
 - Anode fouling eliminated

- Improved coating adhesion in as-deposited state
- Coating deposition rate appears linear
- Maintain coating thickness upon spinel conversion





Varying Coating Thickness

~ 10 μ m coating ~ 2 μ m Cr₂O₃ ~ $21\mu m$ coating ~ $3\mu m Cr_2O_3$



~ 33 µm coating



5µm

SE1

1144

SE1



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Varying Cobalt Concentration



- Minor Cr diffusion
- Minor Fe diffusion

- Minor Cr diffusion
- Some Fe diffusion

- Negligible Cr diffusion
- Fairly significant Fe diffusion

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750 Hour Thermal Soak at 800 °C



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750 Hour Thermal Soak at 800 °C

2 hr. thermal treatment in air atm prior to thermal soak



Oxygen Ka1, Manganese Ka1, Chromium Ka1, Iron Ka1, Cobalt Ka1

 2 hr. thermal treatment in H₂ atm prior to thermal soak



750 Hour Thermal Soak Testing

The ASR is $\leq 20 \text{ m}\Omega \text{ cm}^2$ after 750 hrs. at 800 C

	Sample No.	Thickness (µm)	Atomic%		ASR (mΩ·cm ²)
			Со	Mn	
H ₂ atm exposure for	1132	7.5	89	11	13.3
2 hours followed by	1134	10	91	9	16.7
thermal soak for 750 h	1136	13	92	8	9.6
Air atm exposure	1133	12	85	15	13.0
for 2 hours followed	1135	11	85	15	19.5
by thermal soak for 750 h	1137	14	85	15	13.8



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750 Hour Thermal Soak







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Process Scale-up from 25 cm² to 100 cm²



Process Scale-up from 25 cm² to 100 cm²



25 cm² 430 Stainless Steel Interconnect With Gas Flow Fields



- 3 channel serpentine pattern
- Channel width ~ 0.9 mm
- Rib width $\sim 0.8 \text{ mm}$
- Channel depth ~ 0.45 mm

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25 cm² 430 SS Interconnect With Gas Flow Fields



25 cm² 430 SS Interconnect With Gas Flow Fields



Future Work

- Complete thermal soak to 2000 hours for existing samples
- Development, optimization and validation of the FARADAYICSM Electrodeposition Process for 100 cm² interconnects with gas flow field features
- Long-term on-cell performance evaluation of button cells
- Qualification/Demonstration of Interconnect Coating in Single Cell Test Rig under ideal SOFC operating conditions by potential commercial partners
- Continued development of a more comprehensive economic assessment of the electrodeposition coating process as it relates to interconnect manufacturing.

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Acknowledgments

- Briggs White and the entire NETL SECA team
- This material is based upon work supported by the Department of Energy under Award Nos. DE-SC0001023 and DE-FE0006165. Any opinions, findings, conclusions and recommendations expressed in this material are those of the authors and do not necessarily reflect the views of the DOE.
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