Interconnect Alloys
Metallurgy and Manufacturing

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ATI Allegheny Ludlum

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Introduction

- Environmental exposure conditions can lead to oxidation/functional degradation of interconnects and adjacent components
- Alloy and process development to reduce contribution to SOFC performance degradation
Overview

• Phase I results and timeline
• Current focus of Phase II
  – Silicon removal trials
  – Oxidation of commercially available ferritic stainless steels
  – Production of novel ferritic stainless steels
• Ongoing work
Phase I Review
Results to Date - Phase I

- **Timeline**
  - 12 month period
  - Calendar year 2006
- Proof of concept for solid-state silicon removal
- Melting, processing, and testing of concept alloys
- Testing/analysis of commercially available stainless steels
- Testing/analysis of various oxidation-resistant coatings on commercially available stainless steels
Phase II Status
Silicon in Stainless Steels

- Silicon is present in most readily available stainless steels
- By-product of the AOD steelmaking process
- Commercially available stainless steels generally contain approximately 0.5 wt. % silicon
Silicon Removal Trials

- High-temperature pre-treatment with optional chemical component
- Tested using a variety of Fe-Cr stainless steels (T430, T439, T441HP™ alloys)
- Formation/removal of an SiO$_2$ surface film
Effect on ASR - Prior Work

For T430ss samples pre-oxidized in air for 500 hours at 800°C:

- As-annealed
- Chemical cleaning #1
- Chemical cleaning #2
Silicon Removal

- Production of larger test panels
  - Third party testing and analysis
  - Internal evaluation
- Characterization of treated surface
- Oxidation testing
- Long-term electrical evaluation
Post-Treatment Characterization

T430 post-treatment samples
AES analysis with sputter depth-profiling
Effect of Substrate Thickness

• Thermally activated process
• More effective for thinner samples
  – Absolute quantity of Si removed is a function of temperature, surface area
  – Amount of silicon available for removal (reservoir) is a function of substrate thickness

• Evaluation
  – Rolling trial (0.08-0.15 mm thick T430 samples)
  – Calculations
Effect of Substrate Thickness

starting with an “infinite sheet”

Establish volume element
Set surface area = 1
2w^2 = 1
V_M = w^2 \rho_M = 0.5x = M_M / \rho_M
Effect of Substrate Thickness

Allow the sample to oxidize on both sides

\[ V_{ox} = 2w^2t_{ox} = \frac{M_{ox}}{\rho_{ox}} \]

\( M_{ox} \) allows for the determination of Si consumption / residual \( M_{Si} \) in the volume element after the formation of an oxide layer of thickness \( t_{ox} \)
Effect of Substrate Thickness

Based on trial oxide thickness

Residual silicon (w/o): 0.3% Si, 0.2% Si, 0.1% Si

Substrate thickness (mm): 0.00, 0.10, 0.20, 0.30, 0.40, 0.50

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Oxidation Testing

- Oxidation testing carried out in a variety of environments
  - Ambient air
  - Air + 10% water vapor
  - 4%H₂ + 10% water vapor in argon carrier
- Testing using duplicate specimens at 800°C
  - T430, T430 De-Si
  - T439, T439 De-Si
  - T441HP™ alloy, T441HP De-Si
  - E-BRITE® alloy
## Alloy Compositions

<table>
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<tr>
<th>Element</th>
<th>T430 S43000</th>
<th>T439 alloy S43035</th>
<th>T441HP™ alloy S44100</th>
<th>E-BRITE® alloy S44627</th>
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<td>0.015</td>
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Oxidation Testing

- Type 430 exhibited good oxidation resistance in ambient air but one sample (desiliconized) exhibited breakaway oxidation in humidified air.
- Type 439 exhibited a consistent tendency for spallation in the as-received condition. Spallation did not occur in desiliconized Type 439.
- T441HP™ alloy exhibited the best general resistance to oxidation.
Oxidation Testing

- E-BRITE® alloy exhibited a tendency towards weight loss in humidified air, which is consistent with past work (this alloy was not tested in ambient air).
- The exposure to simulated anode gas (Ar-H$_2$-H$_2$O) was the least aggressive of all of the test exposures. De-siliconization was uniformly beneficial in the SAG composition tested.
Oxidation Test Results

Weight Change (mg/cm²) vs. Time (h)

- T-430 430K
- T-430 430L
- T-430 Desiliconized 430E
- T-430 Desiliconized 430F

Ambient air, 800°C
Oxidation Test Results

Ambient air, 800°C
Oxidation Test Results

Ambient air, 800°C
Oxidation Test Results

Air + 10% water vapor, 800°C
Oxidation Test Results

Air + 10% water vapor, 800°C

Weight Change (mg/cm²)

Time (h)

T-439 439G
T-439 439H
T-439 Desiliconized 439A
T-439 Desiliconized 439B

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Air + 10% water vapor, 800°C
Oxidation Test Results

Air + 10% water vapor, 800°C

Weight Change (mg/cm²) vs. Time (h)

- T-441 441G
- T-441 441H
- T-441 Desiliconized 441A
- T-441 Desiliconized 441B
Oxidation Test Results

Air + 10% water vapor, 800°C

[Graph showing weight change over time for E-BRITE alloy EBRITEA and E-BRITE alloy EBRITEB]
Oxidation Test Results

![Graph showing weight change over time for different materials under SAG, 800°C conditions.]

- T-430 430I
- T-439 439I
- T-441 441I
- T-430 Desiliconized 430C
- T-439 Desiliconized 439C
- T-441 Desiliconized 441C
- E-BRITE alloy EBRITEC
Novel Ferritic Stainless Steels

- Currently processing a set of lab-melted VIM heats
- Optimized compositions
  - High- and low-chromium variants
  - Minor element control
  - Advanced processing
Summary - Alloy Characterization

- Oxidation screening testing is complete
- Long-term oxidation testing ongoing
- ASR evaluation of a matrix of alloys has been initiated
Summary - Current Development

• Compositional matrix melted and processed to sheet
• Silicon removal trials ongoing
Acknowledgements

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Evaluation of a Functional Interconnect System for Solid Oxide Fuel Cells

• Lane Wilson, Wayne Surdoval, Ayyakkannu Manivannan (PM)
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