Durable, Impermeable Brazes for Solid Oxide Fuel Cells

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Outline

• Background and Motivation
  • Benefits of Silver-Copper Brazes
  • Problems with Silver-Copper Brazes
  • Proposed Strategy

• Results and Discussion
  • Partially Sintered Ni Layers
  • As Brazed Joint Microstructures
  • Oxidized Braze Joints
  • Mechanical Properties with/without Oxidation
  • Dual Atmosphere Isothermal Test
  • Dual Atmosphere Thermal Cycling Test
  • Other Applications

• Conclusions
Conventional Reactive Air Brazes Has Many Benefits

- CuO improves the wetting properties
- Brazing can be performed in air
- No flux is needed
- Can be used on a variety of ceramics
Reactive Air Brazes Have Several Fatal Flaws

Braze joint will be exposed to dual atmospheres (H₂/Air) in SOFC operation.

1. Reactive air silver brazes are only partially wetting, resulting in occasional manufacturing defects (Type I Pores);

2. Reduction of reactive air additions (CuO) by hydrogen during SOFC operation can result in Type II Pores;

3. Type III pore formation due to H₂ and O₂ reaction. CuO additions do not prevent the formation of Type III Pores produced when hydrogen and oxygen dissolved in the braze meet and form water pockets.

Hypothesis: Porous Nickel Layers Can Be Used Instead of Reactive-Air Elements

- Ni will not melt before Ag;
- Ni will not dissolve in Ag;
- The Ag wetting angle on Ni is \( \sim 30^\circ \) in inert atmosphere.

Porous Nickel Layers Could Lead to Increased Braze Lifetimes

Braze joint will be exposed to dual atmospheres (H₂/Air) in SOFC operation.

1. **No Type I Pores** with improved wetting characteristics;

2. **No Type II Pores** since no oxides will form during brazing;

3. **Delayed onset of Type III pore formation** without Type I and Type II pores

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Porous Ni Interlayer Fabrication

Screen Print Ni Paste onto YSZ Substrates

Partially Sintered Ni Interlayer on YSZ

Ni Paste

Organics

YSZ

Pre-sinter

900°C, 2 hrs

5 °C/min

Ar Flow at 20 sccm

5 °C/min

Pre-sinter

1 inch

Porous Ni Interlayers with Uniform Thickness

- The screen-printed, pre-sintered Ni layer has a uniform thickness of ~20 µm on the YSZ.
- There are 2~6 particles through the thickness of the porous layer.

As Brazed Joint Microstructure
Brazing Set-up

1000°C, 15/30 mins

5 °C/min

Ar Flow at 20 sccm

5 °C/min

Stainless Steel

Ag Foil

Porous Nickel

Yttria-Stabilized Zirconia

NiO|Yttria-Stabilized Zirconia


Ag-Ni Brazing Eliminates Type I Pores

As Brazed Sample

1 mm

- Solid, dense joints were achieved with the Ag/Ni method using Ag foils.
- Whereas organics (binder) used in the paste form of braze filler materials often lead to large pores.

Ni Relocates to the Stainless Steel Side of the Joint During Brazing

- There is a reaction layer at the SS interface, comprising Ni, Fe and Cr.
- After 30 mins of brazing, the Ni interlayer will be totally transient.

Nickel is Likely Transported via Diffusion and Convective Transport

15 Mins Brazed Sample

30 Mins Brazed Sample

POST OXIDATION
Most of the Joint is Unaffected by Oxidation

As Brazed

After 120 Hours of Oxidation

After 500 Hours of Oxidation

• Samples were held at 750°C in an air furnace for oxidation.

• No obvious porosity developed after 500 hours of oxidation in air.

Most of the Joint is Unaffected by Oxidation

As-Brazed 120 Hour Oxidized 500 Hour Oxidized

Oxidation Occurs at the Stainless Steel Side of the Joint

As-Brazed

120 Hour Oxidized

500 Hour Oxidized

A Protective Chromia Scale Forms Within the Rxn. Layer

120 Hours of Oxidation

500 Hours of Oxidation

MECHANICAL TESTS
Symmetric Double Shear Lap Set-up with Tensile Loading

- Displacement Control: 0.009mm/min ($\sim 10^{-3}$ s$^{-1}$)
- Test to Failure

Tests Show Good Ductility Followed by “Brittle Fracture”

\[ \delta: \text{Displacement per joint}; \quad \sigma_{\text{shear}}: \text{Shear stress}; \quad \varepsilon_{\text{shear}}: \text{Shear strain} \]

\[ a: \frac{1}{4}” \text{ (lateral length of the joints)}; \quad F: \text{Load}; \quad t: \text{Joint thickness}; \quad \delta = \text{Extension}/2; \]

\[ \sigma_{\text{shear}} = \frac{F}{2[a^*(a-\delta)]}; \quad \varepsilon_{\text{shear}} = \frac{(\delta/2)}{t}. \]

Displacement were measured with extensometers.

The Braze Interface Strengths are Both Higher than the Anode Supported YSZ Substrate Strength

- All the joints broke only in the YSZ substrate. The other one has half of the braze/YSZ bonding area detached.

- In some of the joints, the YSZ substrate cracked around the brazed region, indicating the good bonding at the interfaces.


1 cm
DUAL ATMOSPHERE ISOTHERMAL TESTS &
DUAL ATMOSPHERE RAPID THERMAL CYCLING TESTS
Dual Atmosphere Isothermal Test Setup

- Dual atmosphere achieved by flowing 300 sccm of 4% H₂ 96% N₂ through the center hole of the sample stack;
- The assembly was sent up into a furnace to hold at 750°C for 300 hours.

**Dual Atmosphere Test Assembly**

Dual Atmosphere Thermal Cycling Test

- Dual atmosphere achieved by flowing 300 sccm of 4%H₂ 96% N₂ through the center hole of the sample stack;
- Thermal cycling was performed by moving the entire assembly in and out of a three-zone furnace. An average of ~26°C/min heating and cooling rate was applied during the test.

Microstructure of As-brazed Ag₃CuO (RAB) Samples Are Consistent with Literature Reports

Ag₃CuO (wt.) Braze Joint, As-Brazed

Ag₃CuO (RAB) Brazes Show Degradation After 300 hours of Isothermal, 750°C Dual Atmosphere Testing

Ag3CuO (RAB) Braze Showed Similar Degradation After 300 Rapid Thermal Cycles to/from 825ºC

Reminder of the As-brazed Ag-Ni Braze Microstructure

30 Mins Brazed Sample

- There is a reaction layer at the SS interface, comprising Ni, Fe and Cr.
- After 30 mins of brazing, the Ni interlayer will be totally transient.

Ag-Ni Braze Joints Remain Dense After 300 hours of Isothermal, 750°C Dual Atmosphere Testing

<table>
<thead>
<tr>
<th>Through Thickness</th>
<th>SS Interface</th>
<th>YSZ Interface</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Oxidation Side</strong></td>
<td>SS</td>
<td>Ag</td>
</tr>
<tr>
<td></td>
<td>YSZ 20 μm</td>
<td>Ag</td>
</tr>
<tr>
<td><strong>Reduction Side</strong></td>
<td>SS</td>
<td>Ag</td>
</tr>
<tr>
<td></td>
<td>YSZ 20 μm</td>
<td>Ag</td>
</tr>
</tbody>
</table>

0.5 mm

- 5 μm
- Ox
- RE

Through Thickness: Silver

SS Interface: Silver

YSZ Interface: Yttria-Stabilized Zirconia
More Rxn. Layer Oxidation on the RE Side than the Ox Side May be Caused by a NonPassivating Chromia Layer.
Ag-Ni Braze Joints Remain Dense After After 300 Rapid Thermal Cycles to/from 825°C

- Oxidation Side
  - ~74 hours total @ >700 °C
- Reduction Side

Through Thickness

SS Interface

YSZ Interface
OTHER APPLICATIONS
Controlled Wetting/Spreading of Silver and be Used for High Power/High Temperature Circuits/Current Collectors

Zhou et al., Controlled Wetting and Spreading of Metals on Substrates Using Porous Interlayers and Related Articles, USPTO Provisional Patent (Submitted April 17, 2018)
Zhou et al., Controlled Wetting and Spreading of Ag on Various Ceramic Substrates with Porous Ni Interlayers, Scr. Mater, 2018. (In Preparation)
Molten Ag Will Also Infiltrate And Spread Through A Contiguous Porous Ni Pattern

Zhou et al., Controlled Wetting and Spreading of Metals on Substrates Using Porous Interlayers and Related Articles, USPTO Provisional Patent (Submitted April 17, 2018)

Zhou et al., Controlled Wetting and Spreading of Ag on Various Ceramic Substrates with Porous Ni Interlayers, Scr. Mater, 2018. (In Preparation)
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• Conclusions
Quan Zhou et al., Brazing Method Using Porous Interlayers and Related Articles, USPTO Provisional Patent. (Submitted May 10, 2017)
Conclusions

1. A new porous-Ni-enabled Ag brazing approach for improved YSZ-to-stainless steel joining was developed.

2. This technique reduces the porosity commonly found in conventional, dual-atmosphere, Ag-based YSZ-stainless steel braze joints, and hence should produce SOFC braze joints with enhanced lifetimes and operational robustness.

3. Using this new technique, braze joints with good mechanical strength can be produced.

4. The interfacial bonding strength with this new technique is stronger than commercial YSZ substrates, even after prolonged oxidation.

5. Preliminary isothermal aging and rapid thermal cycling tests showed that these brazes are more durable than conventional Ag-CuO brazes.

6. Porous nickel enhanced silver wetting may also be useful for enabling Ag (or other) brazes in other ceramic-ceramic and metal-ceramic applications.
Research Team

• **MSU:**
  - Jason D. Nicholas  PI, now tenured
  - Yue Qi  PI
  - Thomas R. Bieler  PI
  - Quan Zhou  PhD student, now at Hitachi Metals
  - Tridip Das  PhD student, now at Intel
  - Yuxi Ma  PhD student
  - Thanaphong Phongprecha  PhD student
  - Riley O’Shea  Undergraduate student, now at Nexteer Automotive
  - Young Kim  Undergraduate student, now at Fraunhofer USA

• **Delphi:**
  - Rick Kerr (and his team …)
  - Stephanie Surface
  - Bryan A. Gillispie

• **NETL**
  - Joseph Stoffa
Products Resulting From This Work

Ag-Ni Braze and Circuit Pastes


Ag and Ni Alloy Wetting


How D-Orbital Splitting Controls Oxygen Vacancy Polaron Size/Shape, Mobility and Conductivity in the LSF Solid Solution


A New In Situ, Current-Collector-Free, Non-Contact Technique for Characterizing MIEC Materials
Ag Melt Infiltrate the Porous Ni Interlayer and Spread Accordingly

Before

5 °C/min
1100°C, 2 hrs
Ar Flow at 20 sccm

After

5 °C/min
1000°C, 2 hrs
Ar Flow at 20 sccm
**Brazing Set-up**

**Symmetric Double Shear Lap**

- Ni|YSZ 1” × 1”
- Pure Ag
- YSZ

**Brazing for 30 or 15 mins**

1000°C, 15/30 mins

5°C/min, 5°C/min

Ar Flow at 20 sccm

- SS441
- Metal Clips
- Ni|YSZ 1” × 1”
- SS 1” × 1”
- 1 inch

**Al₂O₃ Weights (~50g)**

- Al₂O₃ Support Plates
- Al₂O₃ Spacers
750°C Isothermal Test in Air for Oxidation

Samples were cross-sectioned, polished and examined with scanning electron microscope (SEM) as well as energy dispersive X-ray spectroscopy (EDS).
Inert Atmospheres should be Used as the Brazing Atmosphere

1. Ni won’t oxidize in inert gas (with paste)
2. Inert atmosphere is cost-effective and easy to apply in industrial production
3. Vacuum brazing is not compatible with high-throughput SOFC manufacturing

Potential Problems with the Ag-Ni Brazing System

1. The *volume change* associated with Nickel particle oxidation near the air side of the joint could cause mechanical stress that degrades the joint.
   - This may be OK if a *small amount of Ni* is used in the joint, or if the Ni is transient.
   - Compared to Ag-CuO brazes, compressive stress in the braze (due to the volume expansion accompanying Ni oxidation) is better than Type II porosity (caused by the volume shrinkage accompanying CuO reduction).

2. The *interfacial strength* between Ag/Ni composite and YSZ may be low.

3. The *Oxidation layer* on the stainless steel may reduce wettability.
Project Objective

• **Project Duration:** October 2014 to October 2018

• **2014 Proposal Executive Summary:**

  The objective of the proposed work is to **design and test new, SOFC-compatible, silver-free brazes forming durable, oxygen and hydrogen impermeable protective surface scales**. This will be accomplished using a combined computational-experimental approach to develop new braze compositions with the combination of liquidus temperature, coefficient of thermal expansion (CTE), wetting angle, bond strength, ductility, oxygen conductivity, hydrogen conductivity, and chemical stability necessary to produce SOFCs that can withstand 40,000 hours of 750°C operation and thermal cycling. In addition to 1) instituting a new paradigm for braze development, and 2) generating a wealth of important, fundamental materials property data, the proposed work will provide aspiring DOE SECA Industrial Participant Delphi Inc. with new brazes specifically designed to withstand the extremes in temperature, time, atmosphere and thermal cycling encountered during SOFC operation. These new, self-passivating, non-precious-metal brazes will result in extended SOFC lifetimes and reduced SOFC stack costs.
Hypothesis: Porous Nickel Layers Can Be Used Instead of Reactive-Air Elements

1. Two step approach by
   - Deposit and partially-sinter porous *Ni layers* onto YSZ in Argon
   - Melt silver piece atop the porous nickel layer

2. Ni **will not melt** during brazing (MP$_{\text{Ni}}$=1455°C, MP$_{\text{Ag}}$=961.8°C)

3. The Ni interlayer **will not dissolve** into molten silver.

4. Ag can wet Ni in inert atmospheres, so that molten silver can *melt infiltrate* the Ni interlayer (spontaneous infiltration at wetting angle <50.7°).
   - $\theta = \sim 9^\circ$ in Zr-gettered helium
   - $\theta = \sim 30^\circ$ in inert atmosphere
   - $\theta = \sim 90^\circ$ in air

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ASM handbook Volume 03


The Ag-Ni Braze Has Several Key Benefits

<table>
<thead>
<tr>
<th>Pore Type</th>
<th>Reactive Air Brazing</th>
<th>Ag-Ni Brazing</th>
</tr>
</thead>
</table>
| Type I (wetting) Pore Formation | • $\theta = 45^\circ$ (for Ag-4CuO) occasionally leads to pores during manufacturing [1,2].  
• Organics in the braze paste can also lead to pores during manufacturing [3]. | • $\theta = 30^\circ$ leading to a fully infiltrated porous Ni network [5].  
• Since no organics are used during brazing (these are removed by heating the nickel paste in Ar to obtain the porous nickel network) binder burnout cannot cause pores during brazing. |
| Type II (interfacial) Pore Formation | • With the reduction of CuO along the braze/YSZ and braze/SS interface, micro-pores will form during SOFC operation near the H$_2$ side of the joint [4]. | • Even after 5 days of oxidation a strong, intimate SS-braze joint is maintained (the reaction layer oxides are suspended within the braze and hence do not impact the braze-ss bonding). Also, no reducible oxides form during brazing so that no Type II pores will be formed. |
| Type III (H$_2$+O$_2$) Pore Formation | • H$_2$ and O$_2$ diffuse through Ag and form water pockets (Type III pores) that mechanically compromise the braze joint after $\sim$10,000 hours of SOFC operation [4]. | • Since Type II pores form much faster than Type III pores and thereby provide a short-circuit path for H$_2$ invasion into the center of the braze [6], the elimination of Type II pores can increase joint reliability by delaying the onset of Type III pores. |

Ag-Based Reactive Air Brazes (RAB) Seem Promising at First Glance

1. Reactive air braze additions improve silver on yttrium-stabilized-zirconia (YSZ) and silver on aluminized stainless steel wetting angles.
   - CuO additions to reduce Ag on YSZ in air $\theta$ to $\sim30^\circ$ ($\theta = \sim45^\circ$ for commonly employed Ag-4CuO).

2. Reactive air braze additions improve Ag-Al$_2$O$_3$ and Ag-YSZ bond strengths.

3. Silver reactive air brazing can be done in air.

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Oxygen Diffusivities (Solid Lines) and Hydrogen Diffusivity (Dotted Lines) for Several Common Metals
Ag and YSZ Form a Good Bond with the Assistance of the Ni Interlayer

15 Mins Brazed

30 Mins Brazed

Graph showing the distribution of Ag, Zr, Fe, Cr, Ni, and O over the brazing time.
Inter-Diffusion Occurs at the Stainless Steel Interface

15 Mins Brazed

30 Mins Brazed

Graph showing concentration of Fe, Ni, Cr, and O over the reaction layer.
Minimal Changes at the Ag-YSZ Interface

30 Mins Brazed Sample (As-Brazed, Before Oxidation)

30 Mins Brazed Sample (After 120 Hour Oxidation)

30 Mins Brazed Sample (After 500 Hour Oxidation)
There is a Thin Oxide Layer Developing at the YSZ Interface

**120 Hours of Oxidation**

![Graph showing the composition changes after 120 hours of oxidation.](image)

**500 Hours of Oxidation**

![Graph showing the composition changes after 500 hours of oxidation.](image)
No Compositional Changes Occur within the Ag Upon Oxidation

120 Hours of Oxidation

500 Hours of Oxidation
A Protective Chromia Scale Forms on the Stainless Steel after Oxidation
Vacuum Brazing is Incompatible with SOFC Electrode Materials

- For the **Co rich** compounds, decomposition occurs at a $P_{O_2}=10^{-6}$ bar at 800°C.
- For the **Fe rich** compounds, decomposition was observed at lower $P_{O_2} = 10^{-15}$ bar at 800°C.
- To be safe, brazing should be performed in **inert/air or moderately reducing** atmospheres.

Oxygen nonstoichiometry of $La_{0.6}Sr_{0.4}Co_{1-y}Fe_yO_3-\delta$ ($y=0.2$, 0.4, 0.6, 0.8) as a function of $P_{O_2}$. Closed symbols from high temperature gravimetry; open symbols from coulometric titration.

Hashimoto, S., et al., *Oxygen nonstoichiometry and thermo-chemical stability of La0.6Sr0.4Co1-yFeO3-delta (y=0.2, 0.4, 0.6, 0.8)*. Solid State Ionics, 2010. **181** (37-38): p. 1713-1719.
Ni has good wettability to be infiltrated by Ag

\[
P_C = -(2\sigma_{LV}/r_{eff}) \cos \theta
\]

\[
h^2 = r_{eff}^2 \frac{\Delta P}{4\eta} t \quad (\text{Washburn})
\]

\[
h^2 = r_{eff} \frac{\sigma_{LV} \cos \theta}{2\eta} t
\]

- In equilibrium, infiltration happens when \( \theta < 90^\circ \).

When there’s increase in pore area

\[
h^2 = r_{eff} \frac{\sigma_{LV} \cdot \cos(\theta + \varphi)}{2\eta} \cdot t
\]

- In equilibrium, infiltration happens when \( \theta + \varphi < 90^\circ \).

Ni has good wettability to be infiltrated by Ag

Fig. 4. Cross section of a toroid pore ($R/r = 1$) showing static liquid surface position as a function of contact angle for $\Delta P = 0$ (spontaneous infiltration).

- Through calculation of the ratio between $h$ and $R$ critical wetting angles can be estimated based on packing geometry.
- Smallest critical wetting angle is $50.7^\circ$ for perfect penetration.

Brazing SOFC in Inert Gases is a Lightly Studied Topic

No Formation of Big Type I Pores with Ag Melt Impregnation

15 Mins Brazed Sample

- Solid, dense joints were achieved with the Ag/Ni method using Ag foils.

- Whereas organics (binder) used in the paste form of braze filler materials often lead to big pores.

30 Mins Brazed Sample

Thickness of the Joints

Porous Ni interlayer: ~20 µms
+ 
Ag foil of 75 µms
+ 
Some of the SS elements diffused into the braze

Joint thickness of ~100 µms
Several Strategies for Improving the Air-Tolerance of Ag-Ni Brazes Exist

3. Application of a nano-/meso-porous Ni/Ni-alloy layer (thin film) on the YSZ or SS surface. This can be achieved through chemical dealloying or simple reduction.

Chemically dealloyed Ni$_{33}$Mg$_{67}$ thin film (pure Ni)


Ag foils of 100 μm thickness (produced by Schlenk GmbH, purity: 99.995%) were applied as a simplified standard RAB. The braze foils were cut, chamfered, and

\[
\tau_{\text{Max.}} = \frac{F_{\text{Max.}}}{2L(H - \delta_{\text{corr.}})}
\]

While the total strain

\[
\gamma_{\text{tot.}} = \frac{\delta_{\text{corr.}}}{d_L}
\]

Fig. 5. Stress-Strain curves of as-brazed pure Ag-joints at 550, 675, and 800 °C in air.
A partially sintered Ni particle interlayer has formed on the YSZ surface.

- Sample cross-sectioned to examine the as-sintered microstructure.
- Hot mounted @ ~30kN, ~190°C
No formation of big Type I pores with Ag infiltration
Ag-Ni Braze Optimization

1. Different Ni particle sizes
   - The smallest particle size that can still provide good “wetting” of the Ag on YSZ;

2. Layer thickness of the pre-sintered Ni network
   - The thinnest layer of Ni to achieve similar wetting characteristics;
   - Other technique to apply the Ni layer;
   - Relationship to the formation of the reaction layer at the SS interface.

3. Brazing time and temperature
   - Control of the distribution of Ni (particles/layer);
   - The resulting mechanical properties after oxidation (with different as-brazed microstructures).
Ag-Ni Braze Joint Quality Control

1. Joint Radiography at Delphi ➔ to check for Type I pores
   - With no organics used in the actual brazing step, there should be no Type I pores

2. Test braze joint strengths (tensile tests done here at MSU)
   - Systematic tests to obtain statistically meaningful strength;
   - Bend tests to assess the bonding strength.

3. Test braze joint resistance to rapid thermal cycling at Delphi
   - Again the TMC performance should be similar to Ag-CuO since the matrix is still ductile Ag

4. Test braze joint permeability (at MSU)
   - According the previous literature study, the metallic/braze seal are much better in gas permeability in TMC tests.
   - Also, consider the Type II pores to be suppressed in the long run, gas tightness in operation should be better.
Porous Metal Interlayers for Other Brazing Systems

1. Other two-metal systems
   - Lower temperature systems (like Sn/Al) for lower brazing temperature applications.

2. Incorporate with previous investigated Ni-base brazing systems
   - In the previous work, a Ni-based brazing system with good melting range, excellent oxidation resistance was developed for SOFC applications;
   - The only problem with this Ni-based system is poor wetting on the YSZ, which could be solved with the new porous metal interlayer technique.
Standard Ag-CuO Brazes Have Durability Problems

Cross-section of the brazed section (an extreme case)

- The extremely high hydrogen and oxygen diffusivities in silver ($8 \times 10^{-5}$ and $2 \times 10^{-5}$ cm$^2$/sec at 750° C, respectively), which in long-term operations will lead to:
  - Reduction of copper oxide within the braze ⇒ **Pores** along the interface
  - Formation of steam pockets within the braze ⇒ **Pores** in the matrix, sponge-like structure
- These pores can lead to mechanical failure and hermetic seal failure.

T. Bause, J. Malzbender et. al., Damage and Failure of Silver Based Ceramic/Metal Joints for SOFC Stacks, 2013
Solid Oxide Fuel Cell (SOFC) is a Promising Alternative Energy Technology

[Diagram of SOFC components]

Components and operation of a typical SOFC

Gravimetric and volumetric power densities for various electricity generation technologies.

J. D. Nicholas, “Highlights from the 2013 National Science Foundation Solid Oxide Fuel Cell Promise, Progress, and Priorities (SOFC-PPP) Workshop”
Research Team

• **MSU:**
  • Jason D. Nicholas  Lead PI, SOFC
  • Yue Qi  PI, Computational Materials Science
  • Thomas R. Bieler  PI, Metallurgy
  • Quan Zhou  Graduate Student (Ni-based Brazes)
  • Yuxi Ma  Graduate Student (Cu and Co-based Brazes)
  • Tridip Das  Graduate Student (Simulations)
  • Joe Phongpreecha  Graduate Student (Simulations)

• **Delphi:**
  • Rick Kerr  (and his team …)
  • Stephanie Surface
  • Bryan A. Gillispie