Reactive-Element Free, Silver-Based Brazes for SOFC Applications

Quan Zhou, [1] **Thomas R. Bieler,**[1] **Rick Kerr,**[2] **and** ***Jason D. Nicholas***[1]

[1] Chemical Engineering and Materials Science Department, Michigan State University
[2] Delphi Automotive, PLC

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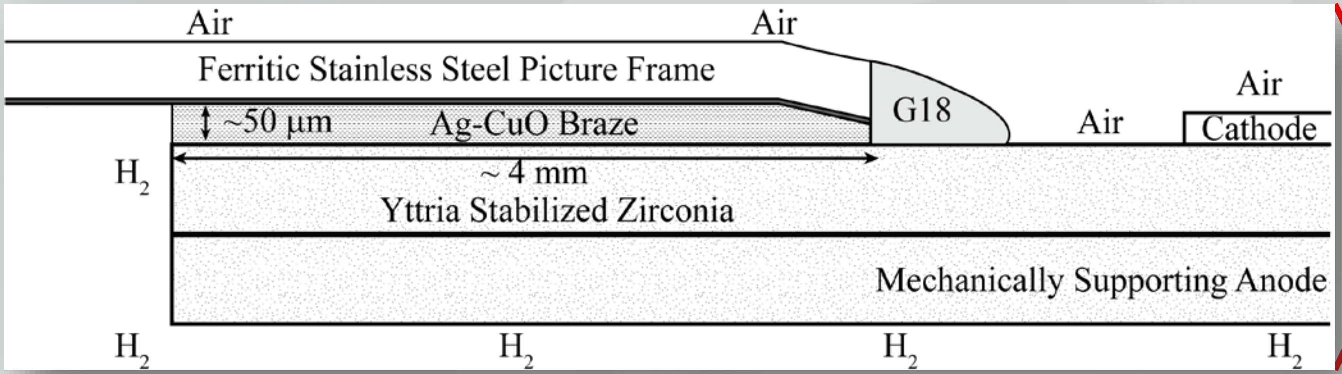
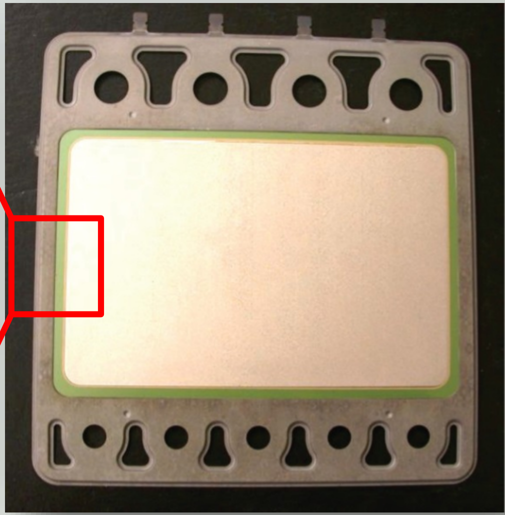
Joe Stoffa, Program Manager

June 13, 2017

Metal Picture Frame Suspended SOFC Schematic

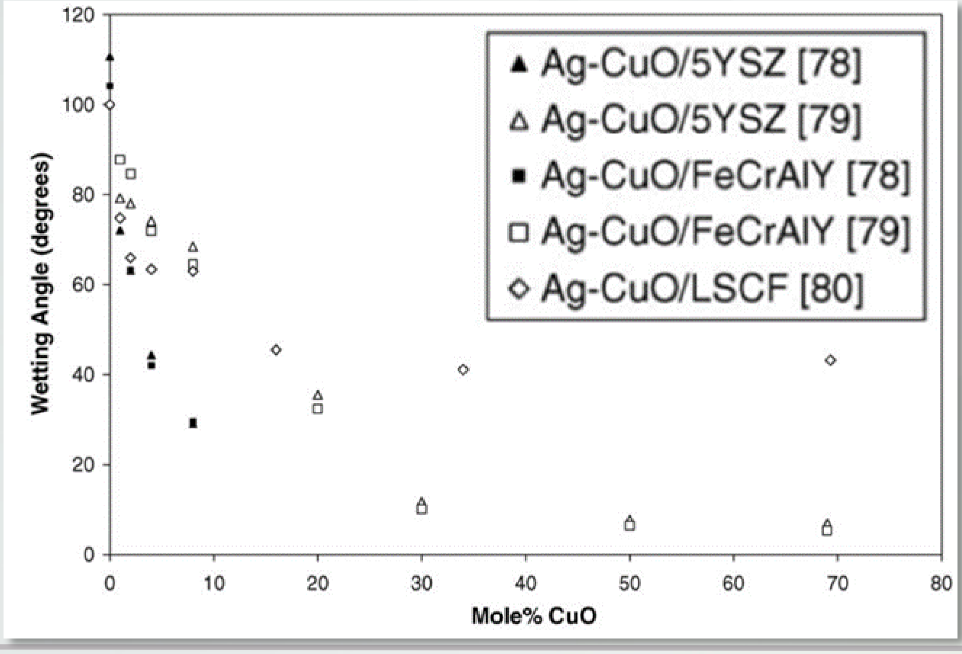


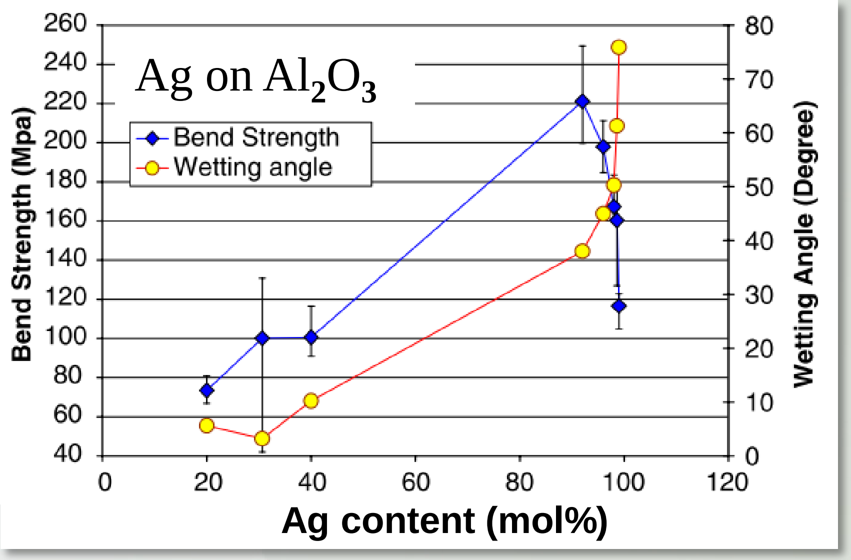


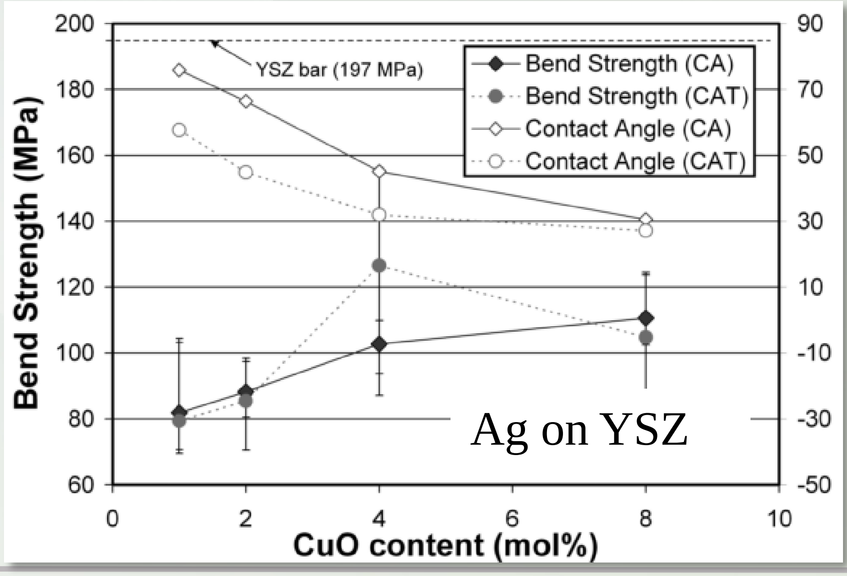
Delphi Technologies Inc, U.S. Patent No US7855030B2

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

1. Reactive air braze additions improve silver on YSZ and silver on aluminized stainless steel ***wetting*** angles.
   - CuO additions to reduce Ag on YSZ in air θ to ~30° (θ = ~45° for commonly employed Ag-4CuO).
2. Reactive air braze additions improve Ag-Al_2O_3 and Ag-YSZ ***bond strengths***.
3. Silver reactive air brazing can be done in ***air***.

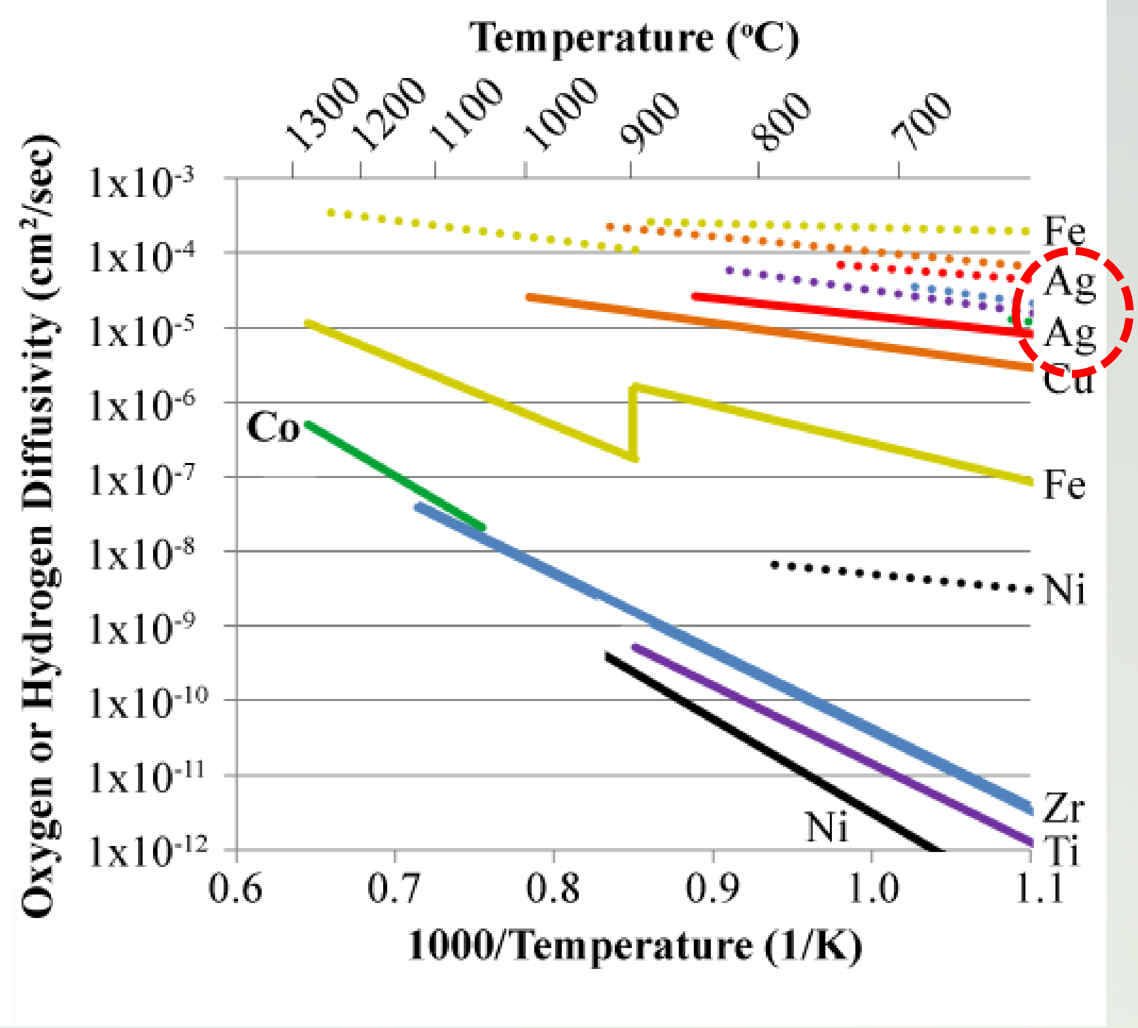






Kim, J.Y., J.S. Hardy, and K.S. Weil, Journal of the American Ceramic Society, 2005. 88 (9): p. 2521-2527.
Kim, J.Y., J.S. Hardy, and K.S. Weil, Journal of Materials Research, 2005. 20 (3): p. 636-643.
Fergus, J.W., Materials Science and Engineering A, 2005. 397 (1-2): p. 271-283.

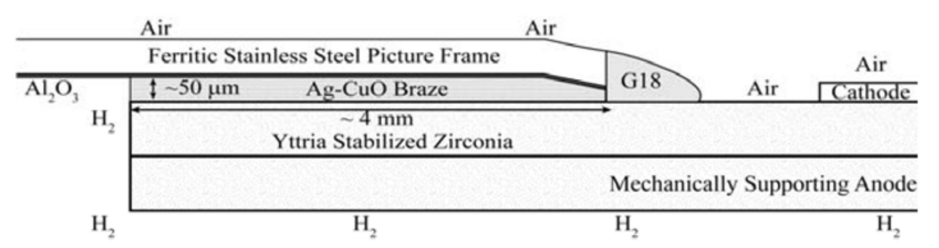
Oxygen Diffusivities (Solid Lines) and Hydrogen Diffusivity (Dotted Lines) for Several Common Metals



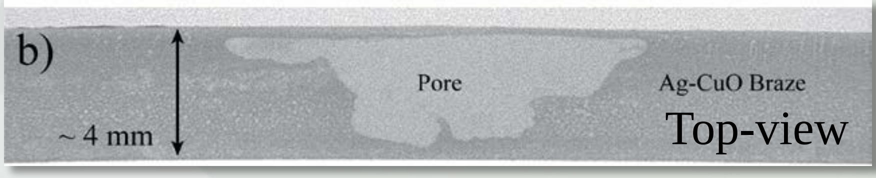
C. Smithells, W. Gale, T. Totemeier. Smithells Metals Reference Book. 8th ed.

Reactive Air Brazes Have Several Fatal Flaws

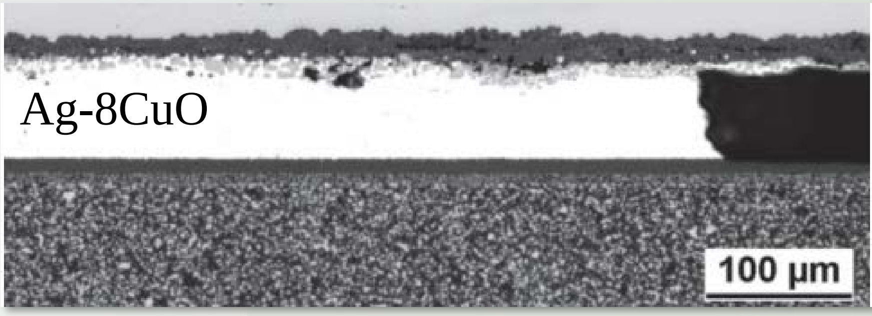
1. Braze joint will be exposed to dual atmospheres (H_2/Air) in SOFC operation.



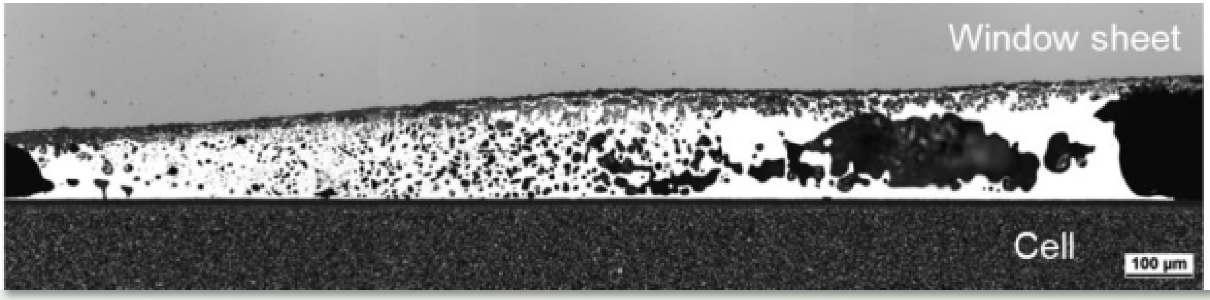
2. Reactive air silver brazes are only partially wetting, resulting in occasional manufacturing defects (**Type I Pores**);



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



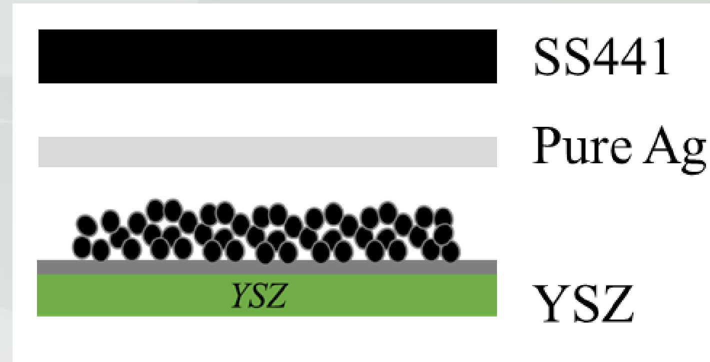
4. **Type III pore** formation due to H_2 and O_2 reaction. CuO additions do not prevent the formation of **Type III Pores** produced when hydrogen and oxygen dissolved in the braze meet and form water pockets.

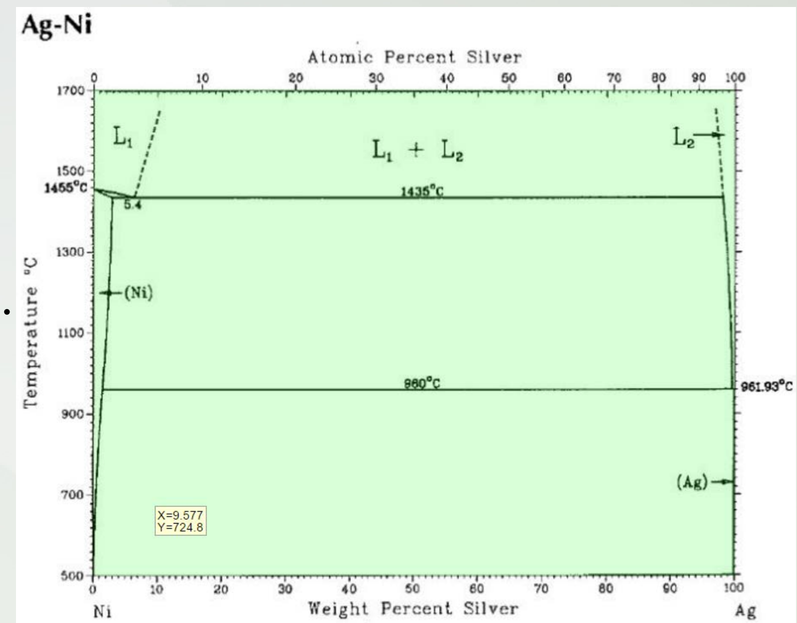


Courtesy of Delphi Automotive.
Bause, T., et al., Damage and Failure of Silver Based Ceramic/Metal Joints for SOFC Stacks. Fuel Cells, 2013. 13 (4): p. 578-583.

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

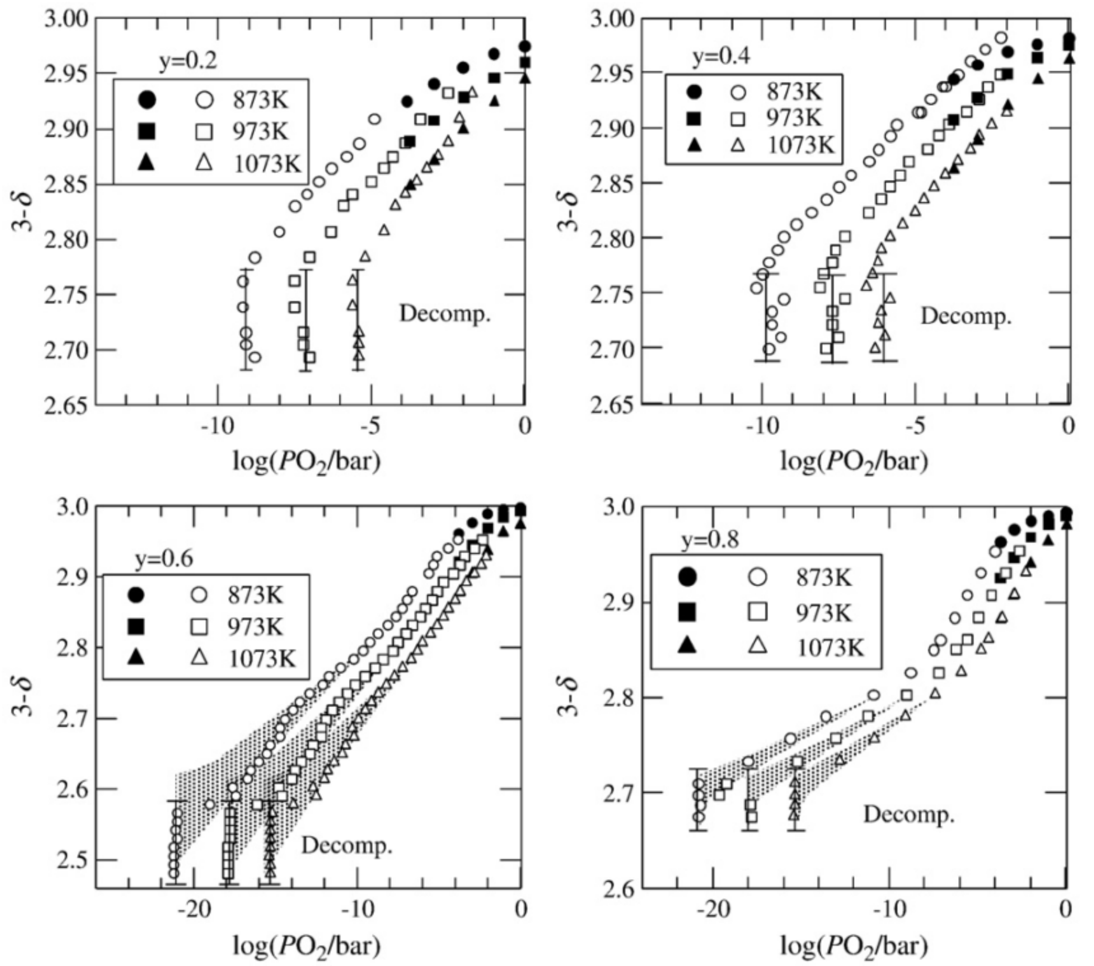
1. Two step approach by
   - Deposit and partially-sinter porous ***<u>Ni layers</u>*** onto YSZ in Argon
   - Melt silver piece atop the porous nickel layer
2. Ni ***<u>will not melt</u>*** during brazing (MP_{Ni}=1455°C, MP_{Ag}=961.8°C)
3. The Ni interlayer ***<u>will not dissolve</u>*** into molten silver.
4. Ag can wet Ni in inert atmospheres, so that molten silver can ***<u>melt impregnation</u>*** the Ni interlayer (spontaneous impregnation at wetting angle <50.7°).
   - θ =~ 9° in Zr-gettered helium
   - θ = ~30° in inert atmosphere
   - θ = ~90° in air





ASM handbook Volume 03
Nagesh, V.K. and J.A. Pask, Wetting of Nickel by Silver. Journal of Materials Science, 1983. ***18*** *(9): p. 2665-2670.*
Hula, R.C., et al., The Wetting Behaviour of Silver on Carbon, Pure and Carburized Nickel, Cobalt and Molybdenum Substrates. Applied Surface Science, 2010. ***256*** *(14): p. 4697-4701.*
Trumble, K.P., Spontaneous infiltration of non-cylindrical porosity: Close-packed spheres. Acta Materialia, 1998. 46 (7): p. 2363-2367.

Vacuum Brazing is Incompatible with SOFC Electrode Materials

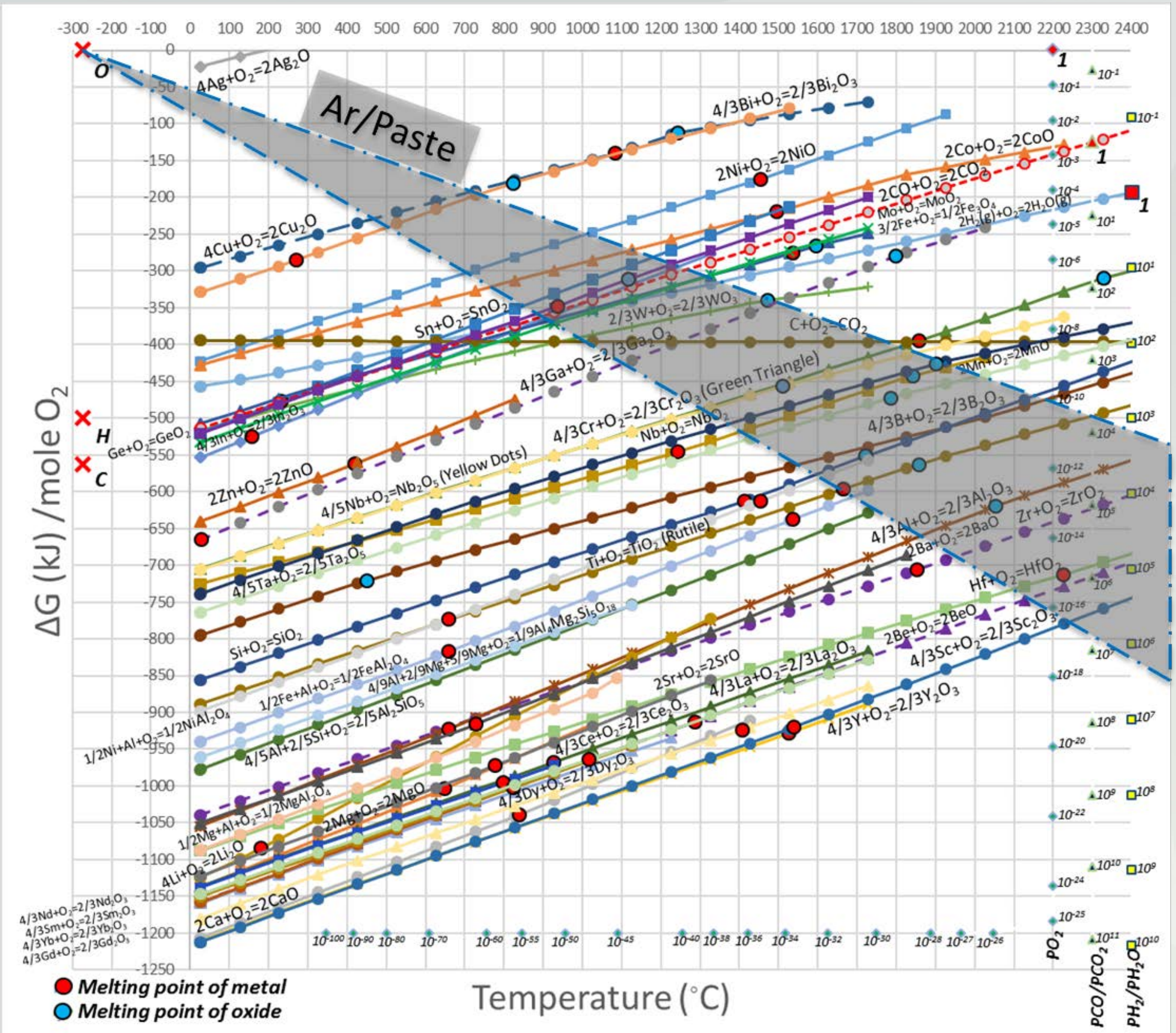


Oxygen nonstoichiometry of $La_{0.6}Sr_{0.4}Co_{1-y}Fe_yO_{3-\delta}$ (y= 0.2, 0.4, 0.6, 0.8) as a function of P_{O2}. Closed symbols from high temperature gravimetry; open symbols from coulometric titration.

- For the ***Co rich*** compounds, decomposition occurs at a P_{O2}=***10^{-6} bar*** at 800°C.
- For the ***Fe rich*** compounds, decomposition was observed at lower P_{O2} =***10^{-15} bar*** at 800°C.
- To be safe, brazing should be performed in ***inert/air or moderately reducing*** atmospheres.

Hashimoto, S., et al., *Oxygen nonstoichiometry and thermo-chemical stability of La0.6Sr0.4Co1-yFeyO3-delta (y=0.2, 0.4, 0.6, 0.8).* Solid State Ionics, 2010. **181** (37-38): p. 1713-1719.

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

Nagesh, V.K. and J.A. Pask, Journal of Materials Science, 1983. ***18*** *(9): p. 2665-2670.*
Hula, R.C., et al., Applied Surface Science, 2010. ***256*** *(14): p. 4697-4701.*

Porous Nickel Layers Could Lead to Increased Braze Lifetimes

Ag-CuO Issues	**Possible Ni-Ag Improvements**
Type I Pores (Wetting)	Ni will improve the wetting characteristics of silver on YSZ.
Type II Pores (CuO Reduction)	Inert atmosphere brazing will avoid Ni oxide formation. (Thus no oxide reduction during H_2 operation)
Type III Pores ($H_2 + O_2$)	Without Type I and Type II pores, Type III pores formation will be delayed.

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).

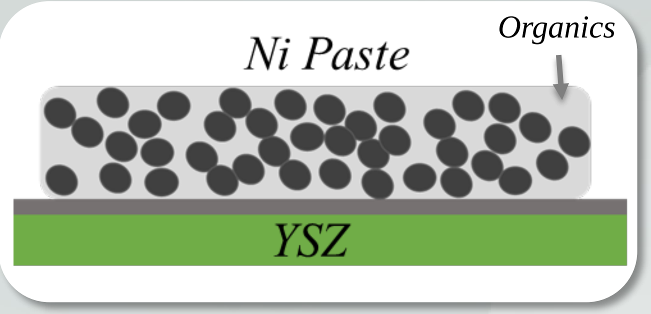
2. The ***interfacial strength*** between Ag/Ni composite and YSZ may be low.

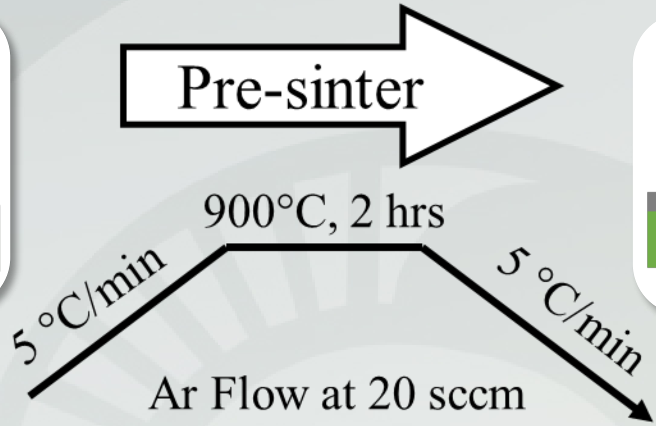
3. The ***Oxidation layer*** on the stainless steel may reduce wettability.

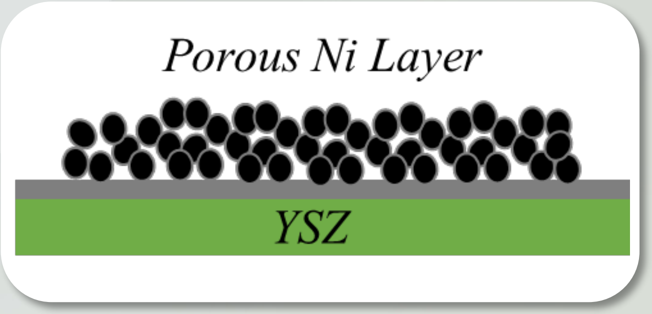
Outline

- **Background and Motivation**
  - Benefits of Silver-Copper Brazes
  - Problems with Silver-Copper Brazes
  - Proposed Strategy
- **Experiments**
- **Results and Discussion**
  - Partially Sintered Ni Layers
  - As Brazed Joint Microstructures
  - Oxidized Braze Joints
  - Mechanical Properties with/without Oxidation
- **Conclusions**

Porous Ni Interlayer Fabrication

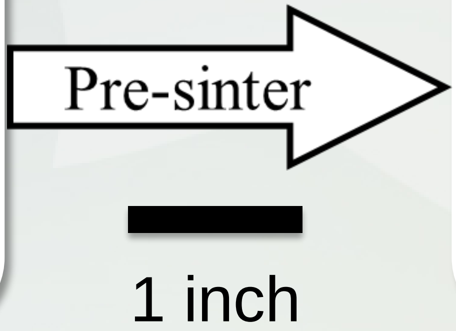






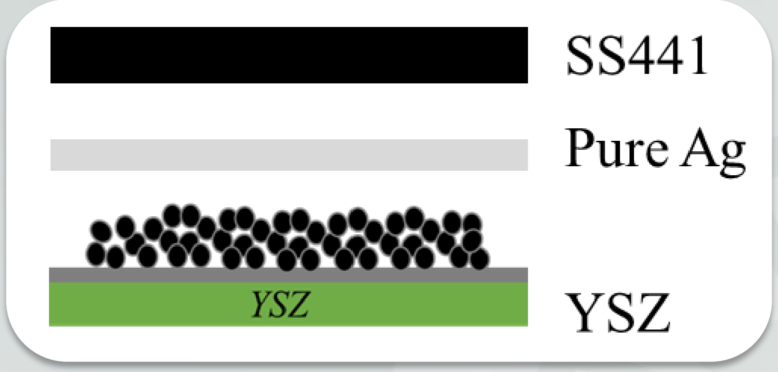
Screen Print Ni Paste onto YSZ Substrates

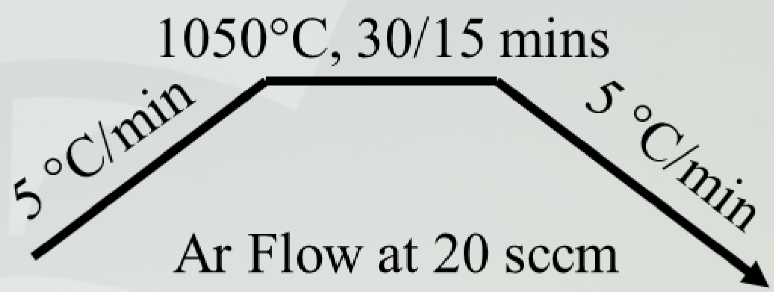
Partially Sintered Ni Interlayer on YSZ



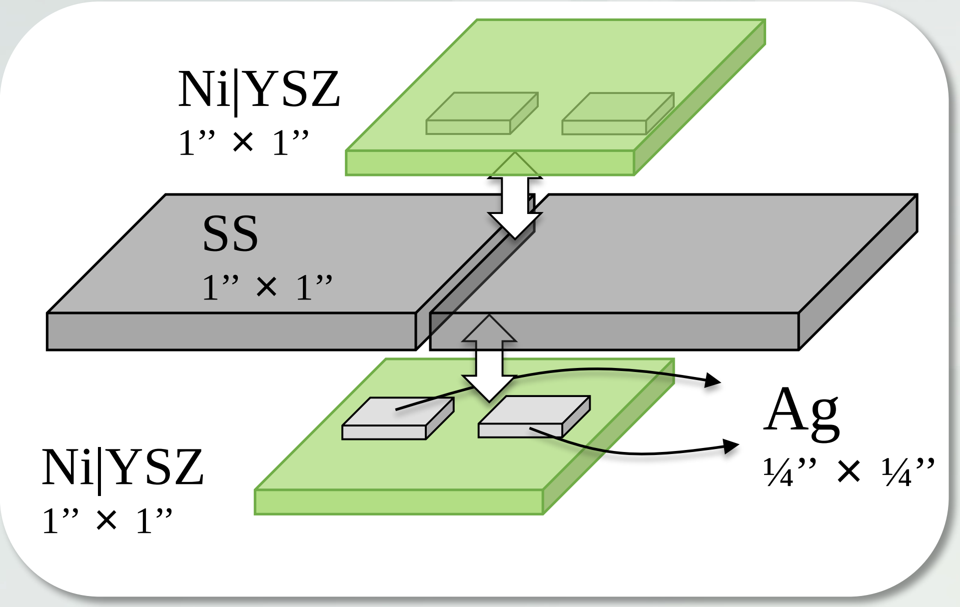
Brazing Set-up

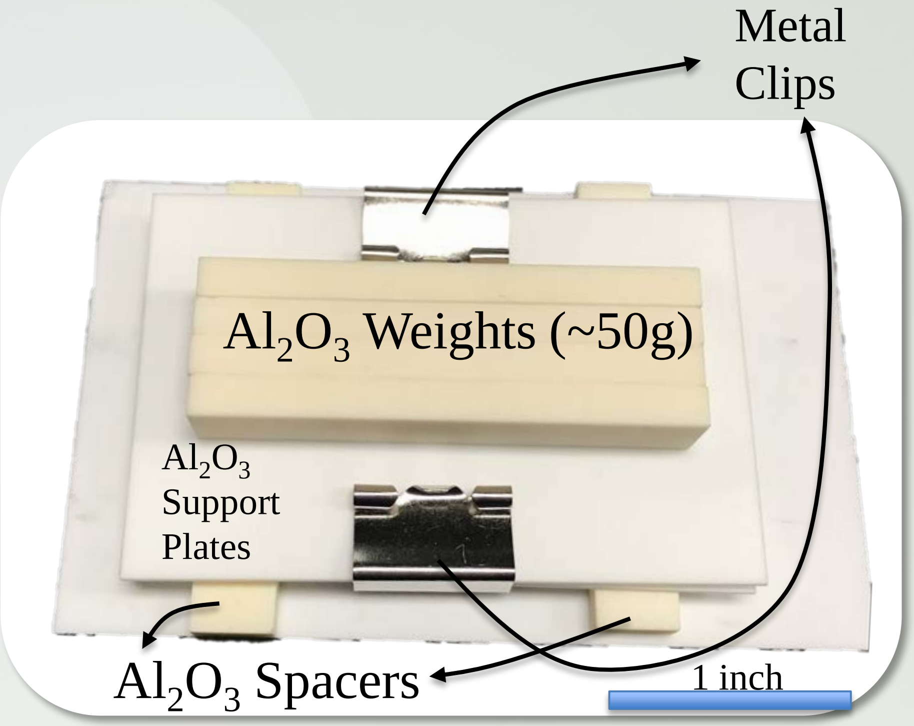
Brazing for 30 or 15 mins





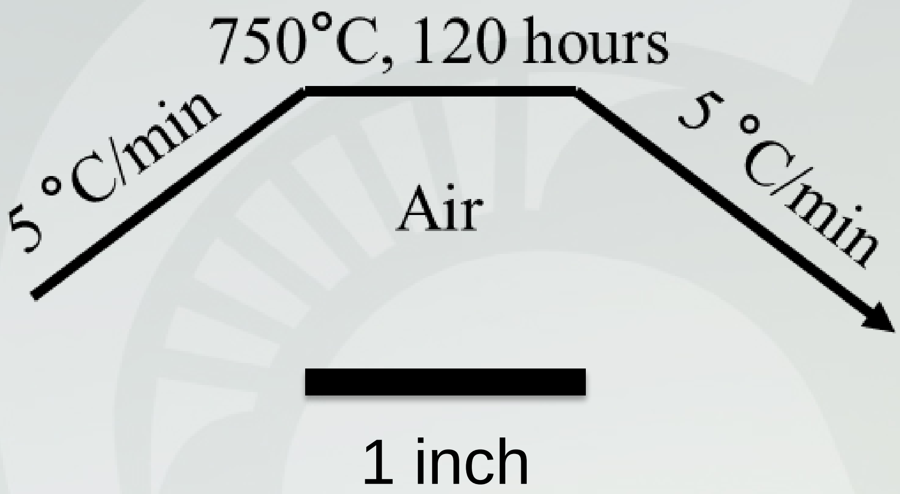
Symmetric Double Shear Lap





Oxidation Studies were Also Performed

As-Brazed



Post Oxidation

Samples were cross-sectioned, polished and examined with scanning electron microscope (SEM) as well as energy dispersive X-ray spectroscopy (EDS).

$L = \sqrt{4Dt} \Rightarrow t = \frac{L^2}{4D} \approx 3150\, sec \approx 0.88\, hrs\ (L\ for\ half\ the\ joint\ width)$

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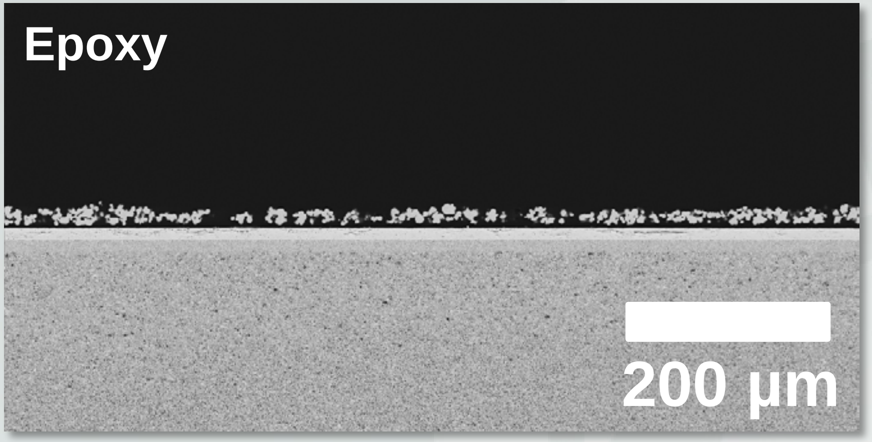
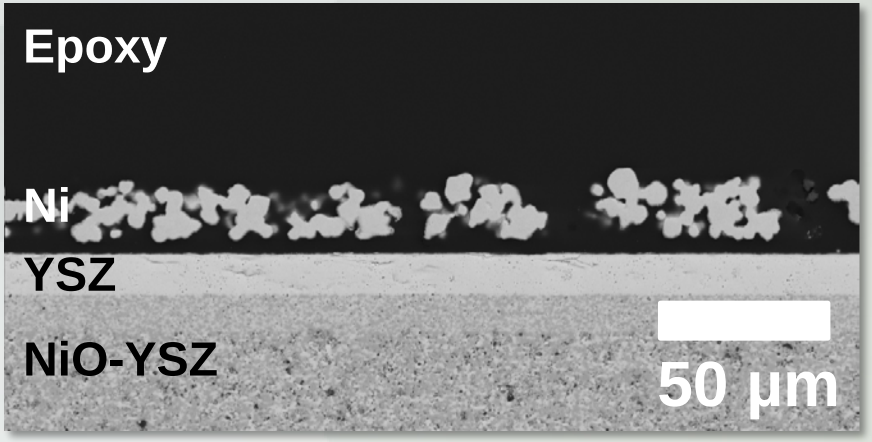
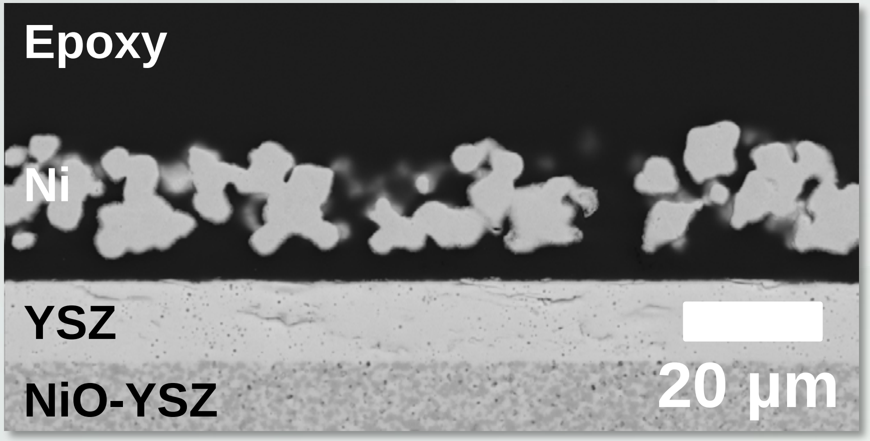
Porous Ni Interlayers with Uniform Thickness is Achieved



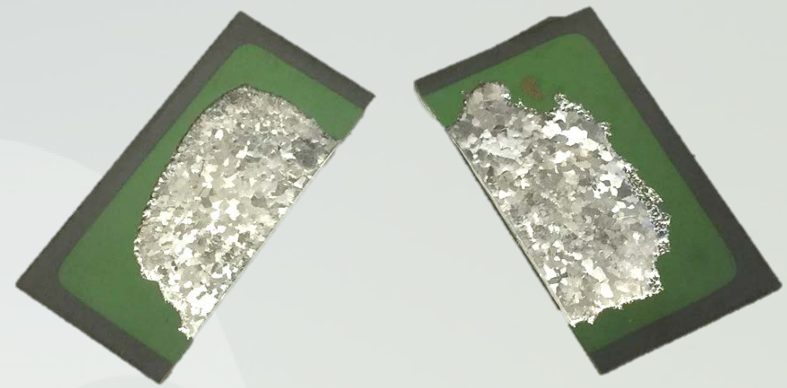




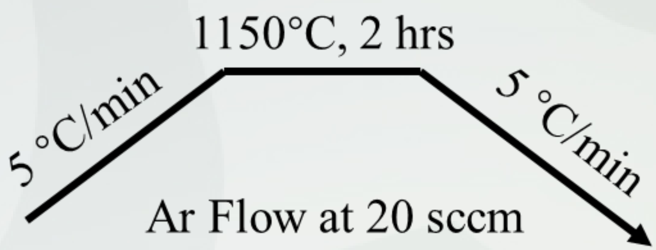
- The screen-printed, pre-sintered Ni layer has a uniform thickness of ~20 µm on the YSZ.
- There are 2~6 particles through the thickness of the porous layer.

Ag Melt Impregnate the Porous Ni Interlayer and Spread Accordingly

Before

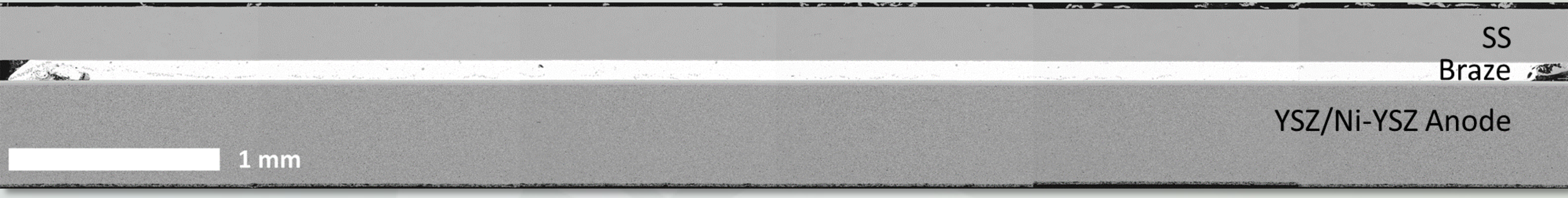


After

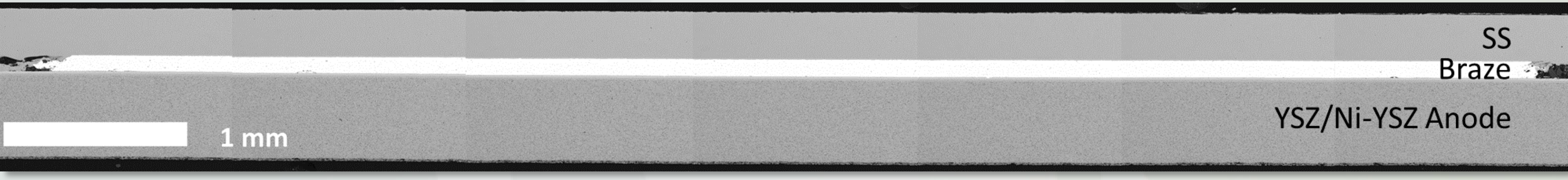


This Brazing Technique Eliminates Type I Pores

15 Mins Brazed Sample



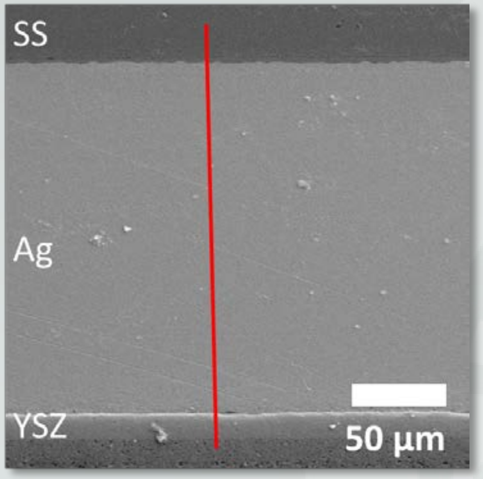
30 Mins Brazed Sample



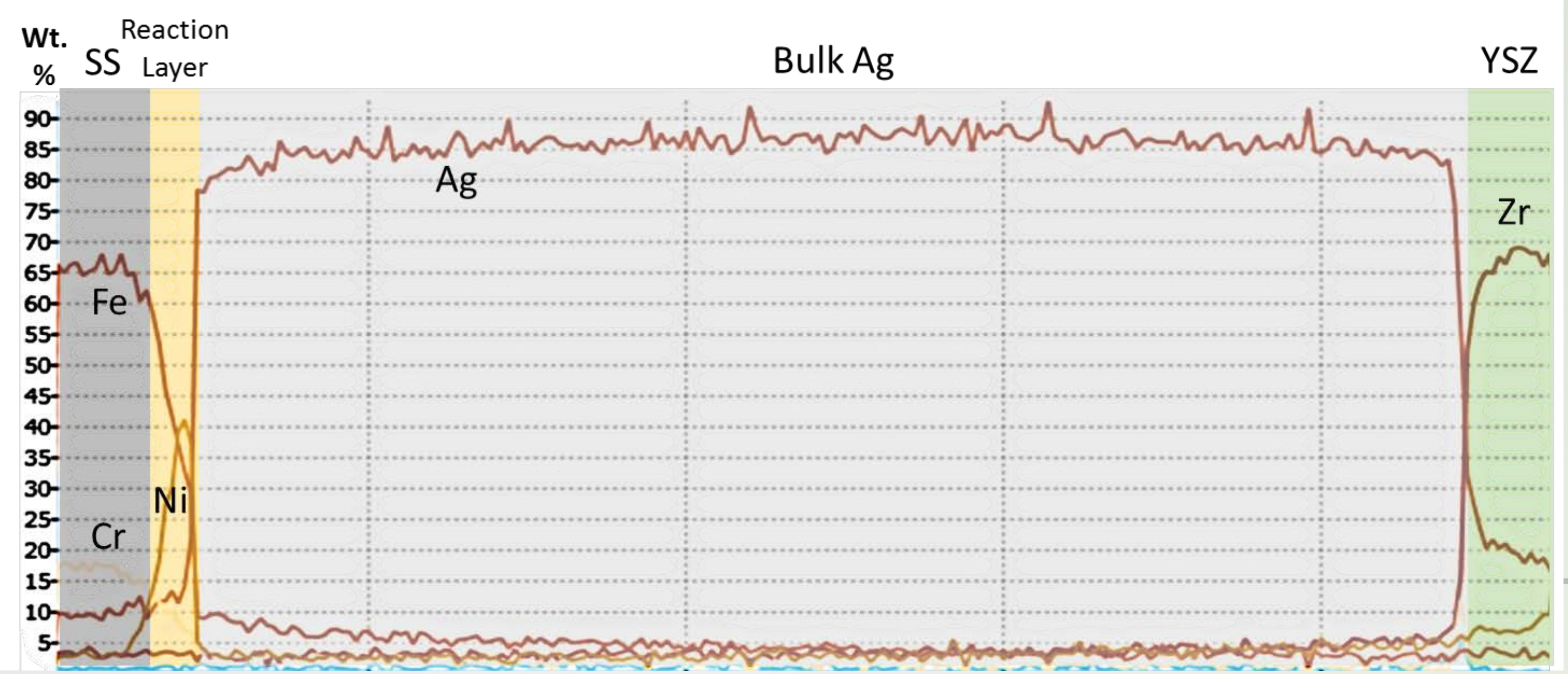
- Solid, dense joints were achieved with the Ag/Ni method using Ag foils.
- Whereas organics (binder) used in the paste form of braze filler materials often lead to big pores.

The Ni Relocates to the Stainless Steel Side of the Joint during Brazing

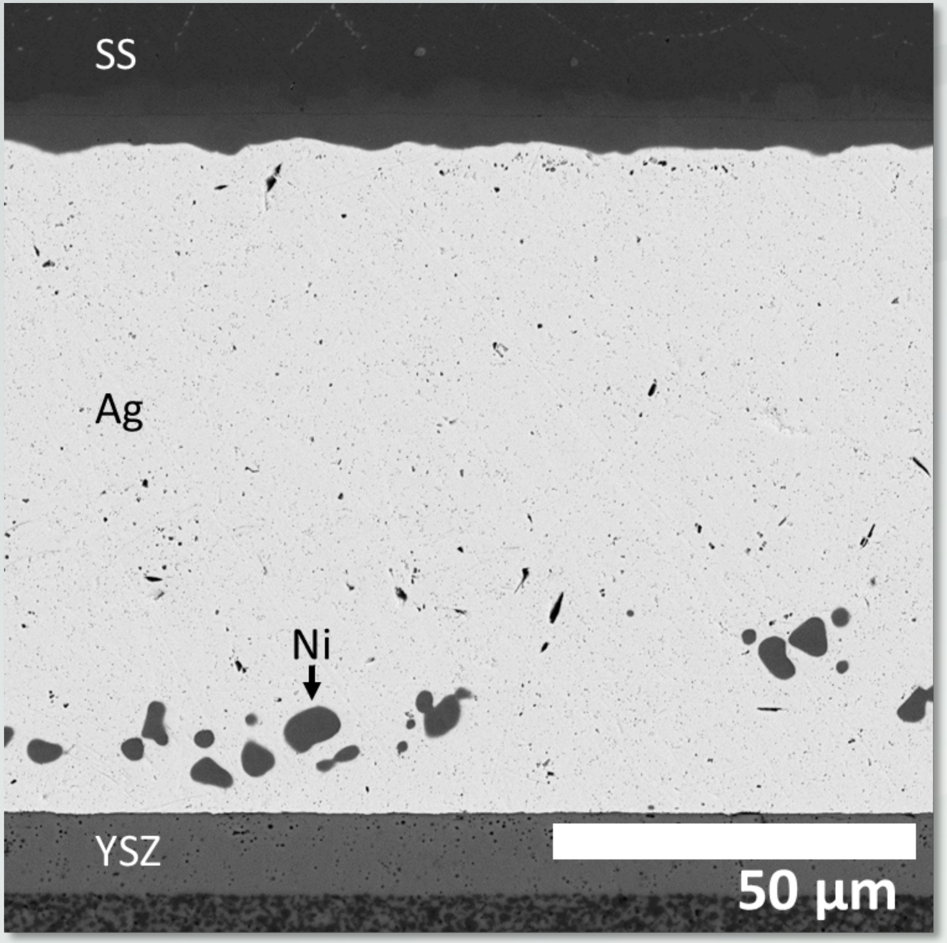
30 Mins Brazed Sample



- 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.
- There is a small amount of Fe and Ni (~5%) in the bulk Ag that's probably due to submicron sized Ni/Ni-Fe particles/precipitates.



Nickel is Likely Transported via Diffusion and Convective Transport



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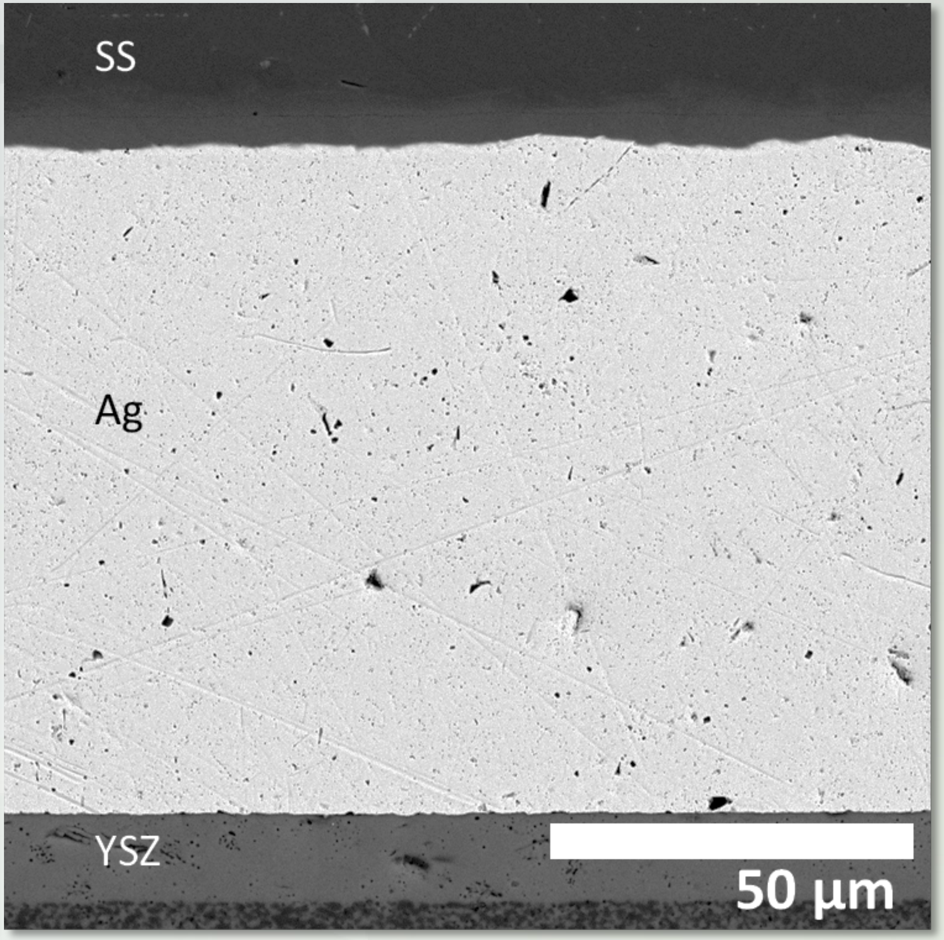
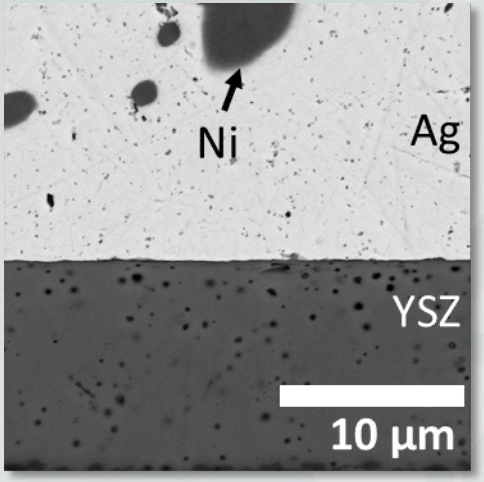
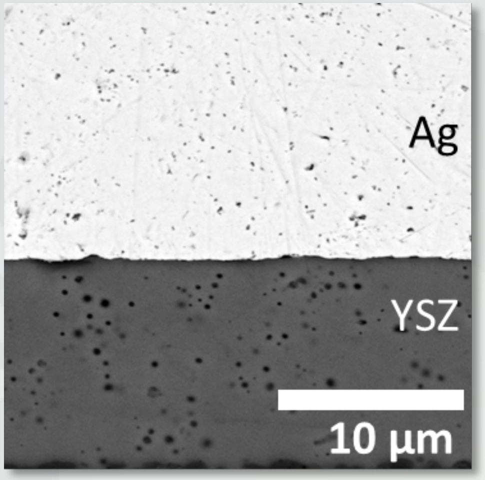


30 Mins Brazed Sample

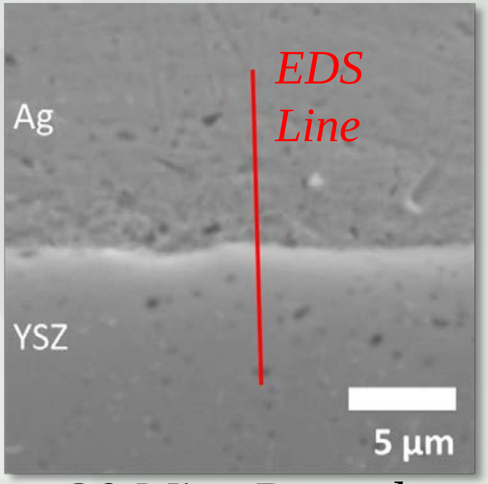
Ag and YSZ Form a Good Bond with the Assistance of the Ni Interlayer



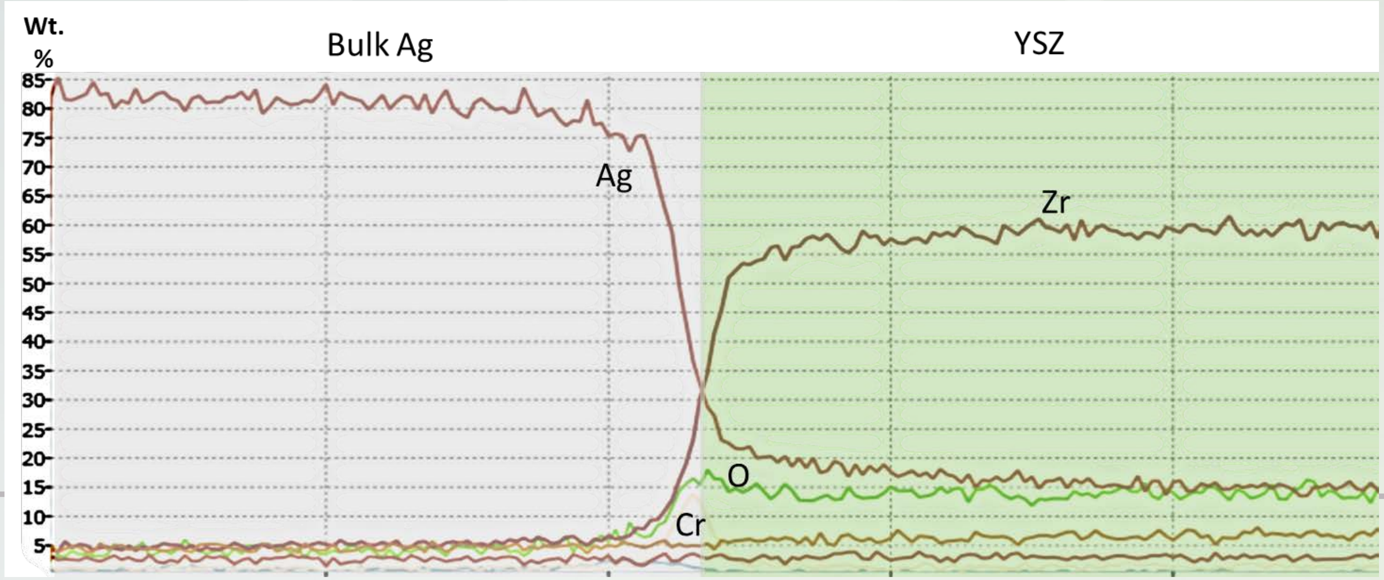
15 Mins Brazed



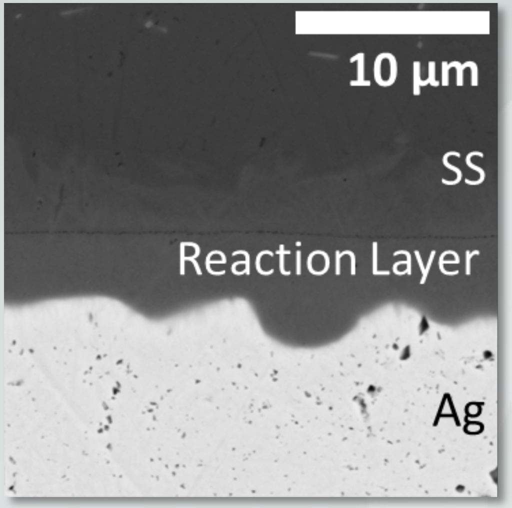
30 Mins Brazed



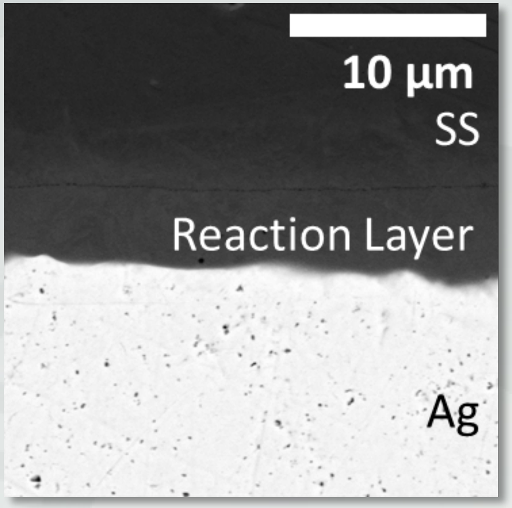
30 Mins Brazed



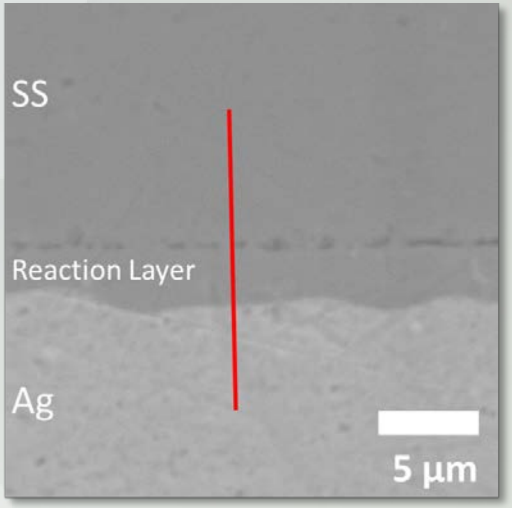
Inter-Diffusion Occurs at the Stainless Steel Interface



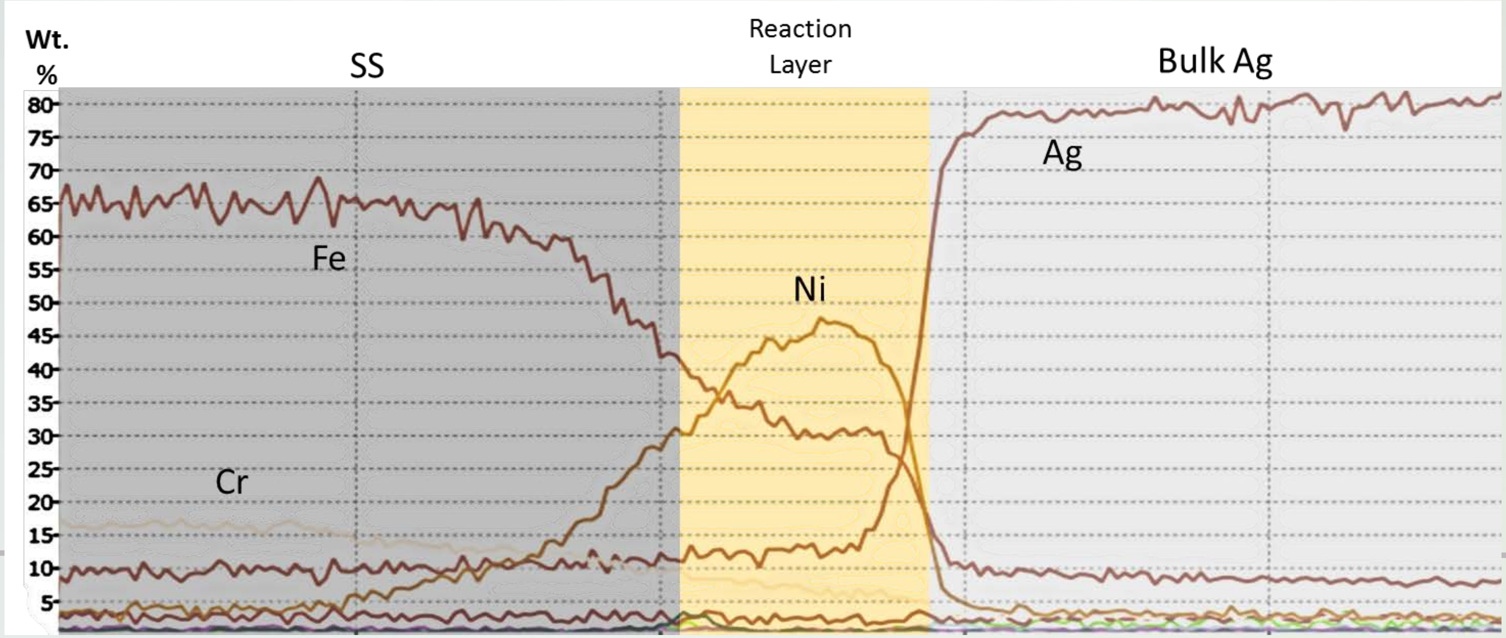
15 Mins Brazed



30 Mins Brazed

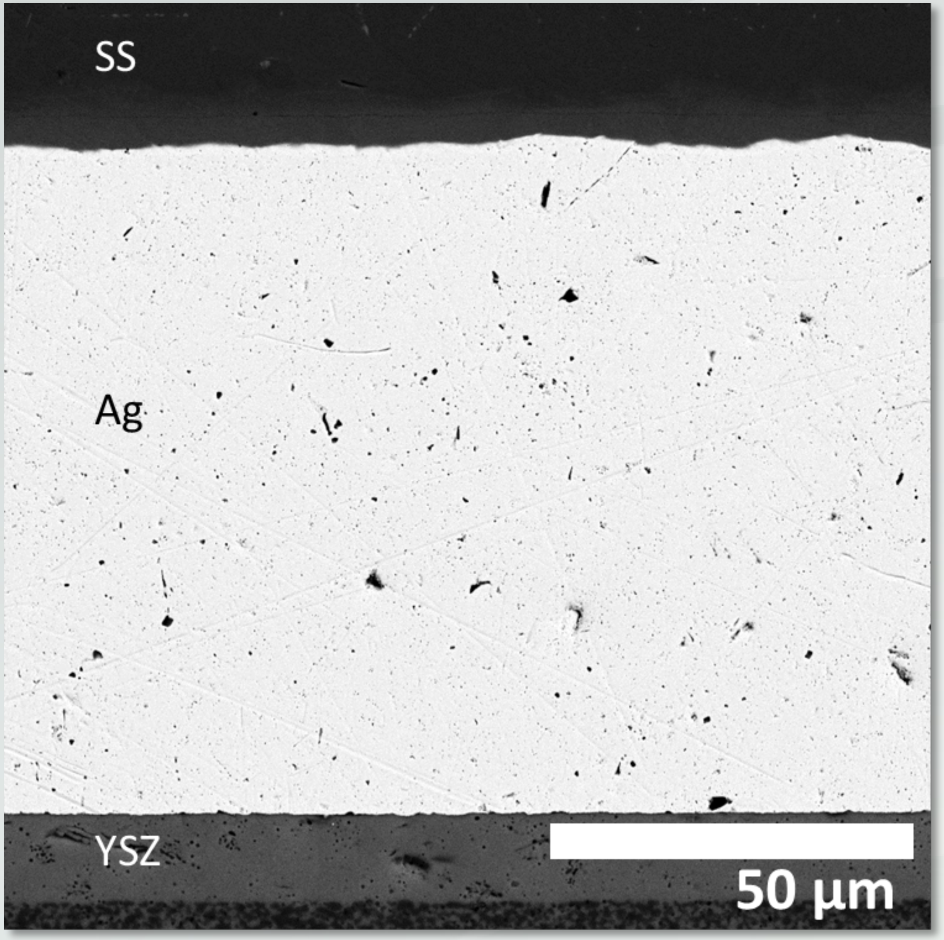


30 Mins Brazed

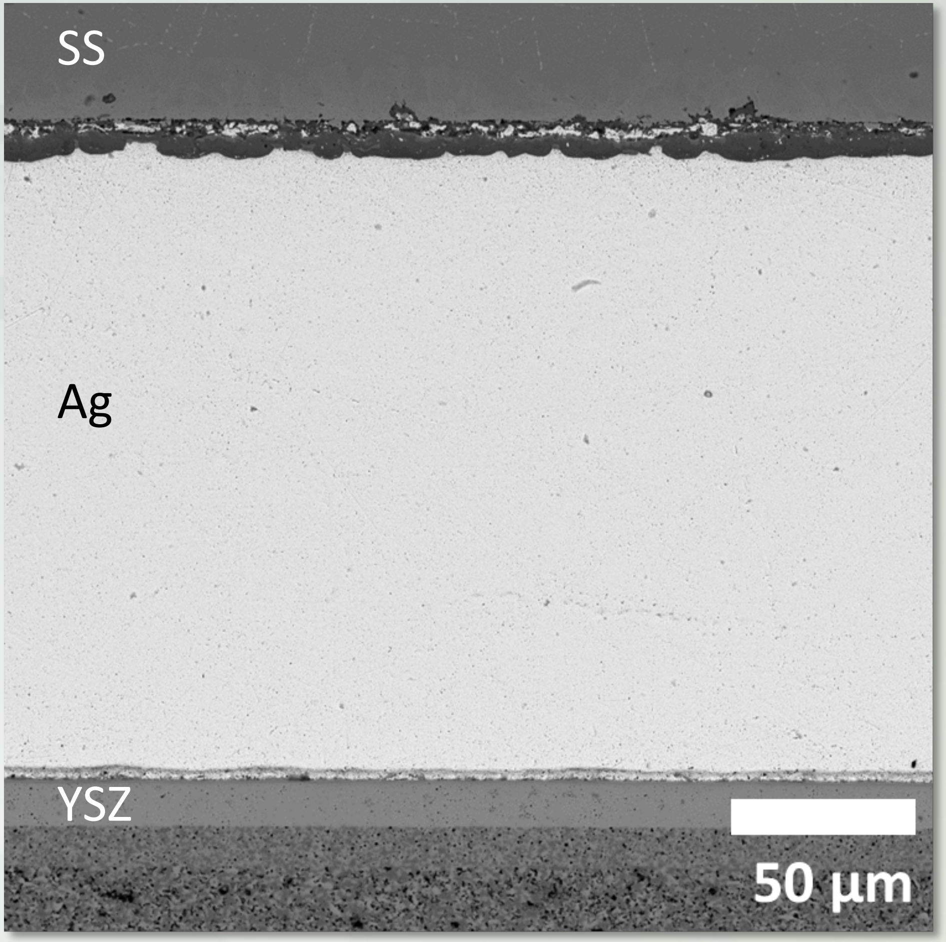


POST OXIDATION

Most of the Joint is Unaffected by Oxidation

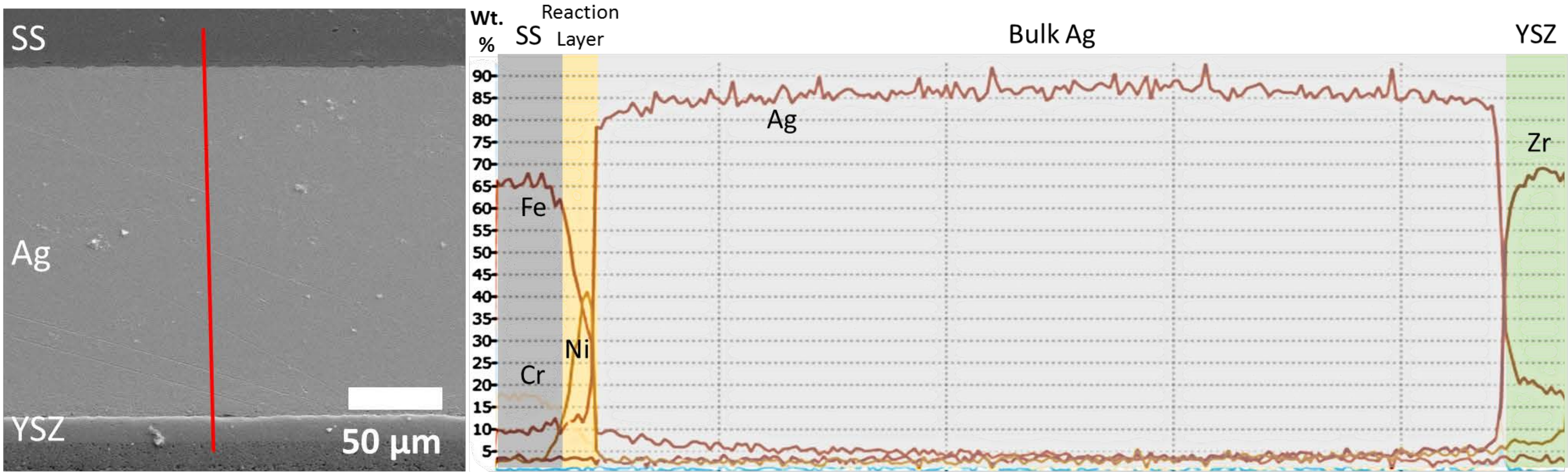


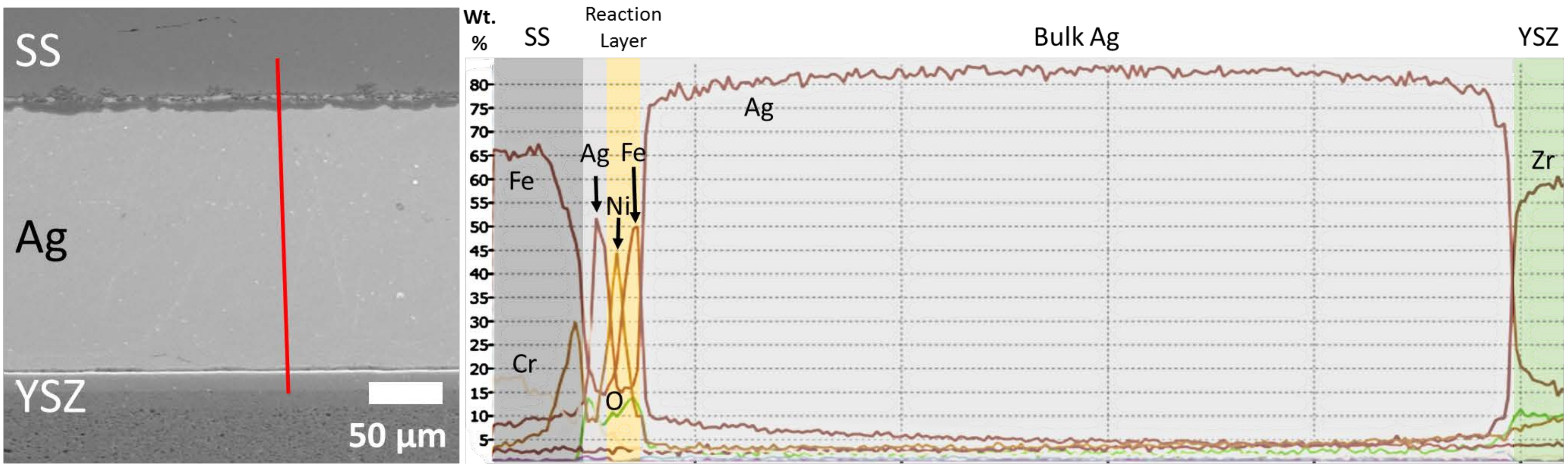
30 Mins Brazed Sample
(As-Brazed, Before Oxidation)



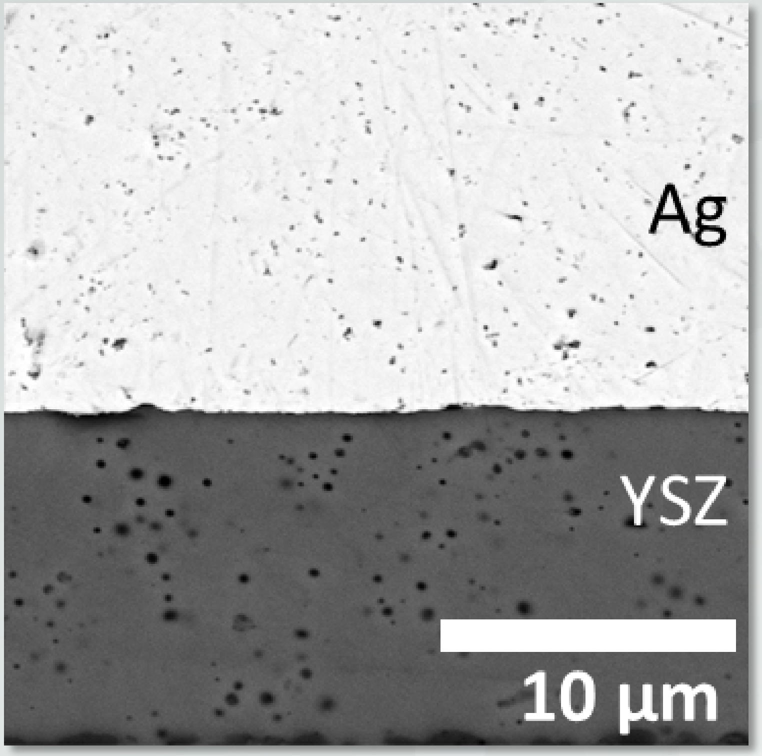
30 Mins Brazed Sample
(After Oxidation)

No Compositional Changes Occur within the Ag Upon Oxidation

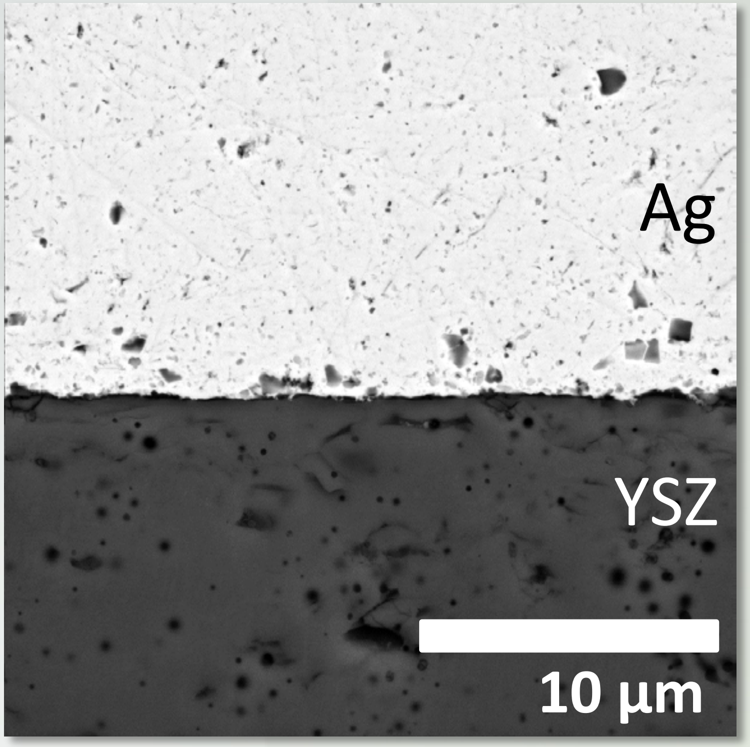




The Ag-YSZ Bond is Unaffected by Oxidation

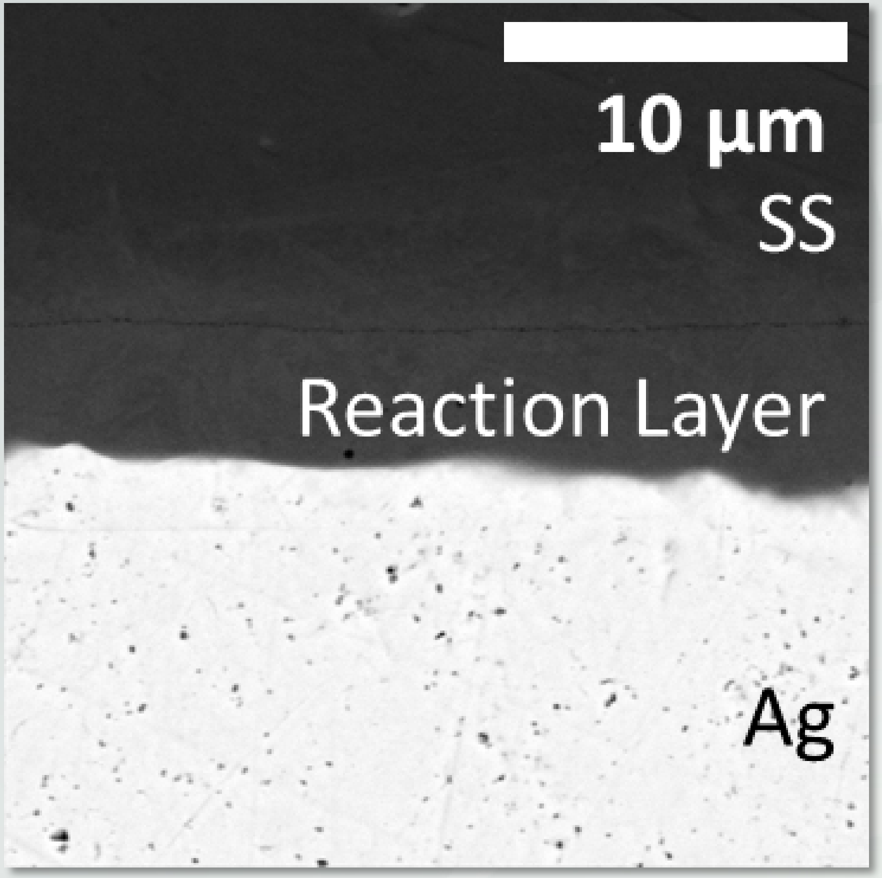


30 Mins Brazed Sample
(As-Brazed, Before Oxidation)

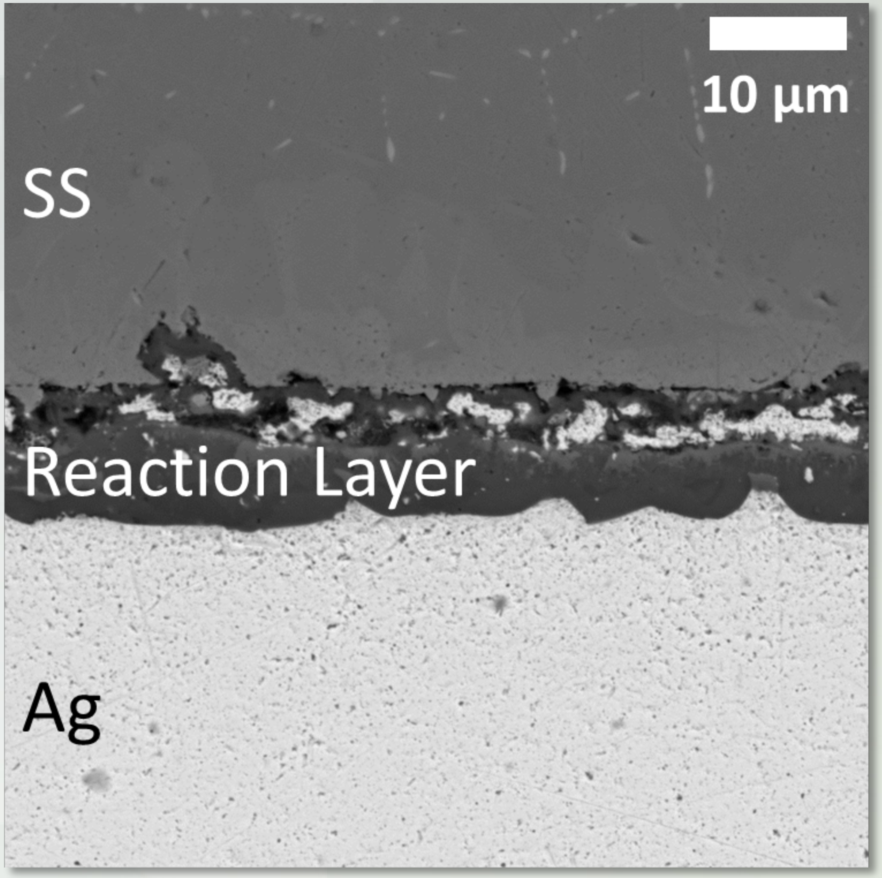


30 Mins Brazed Sample
(After Oxidation)

Oxidation Occurs at the Stainless Steel Side of the Joint

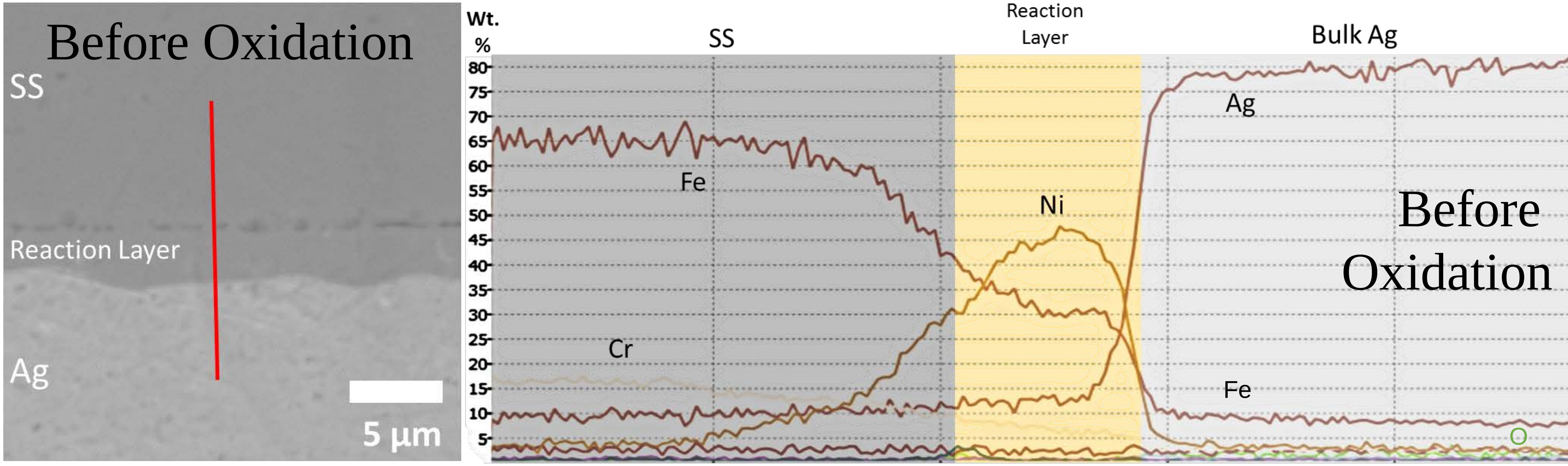


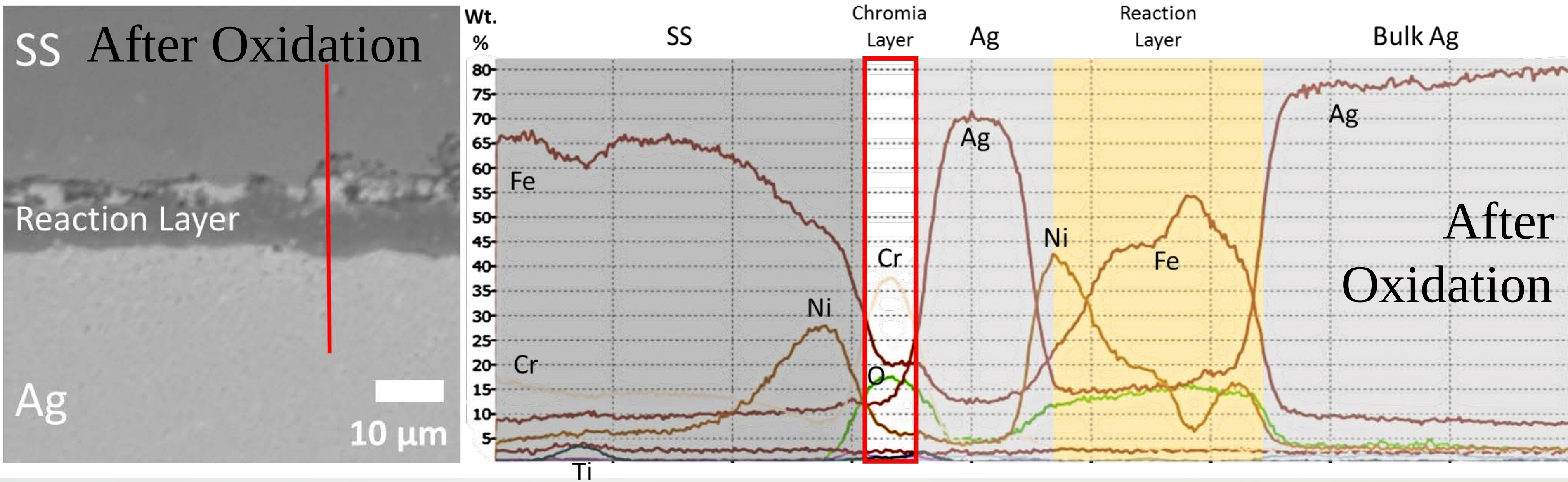
30 Mins Brazed Sample (As-Brazed)



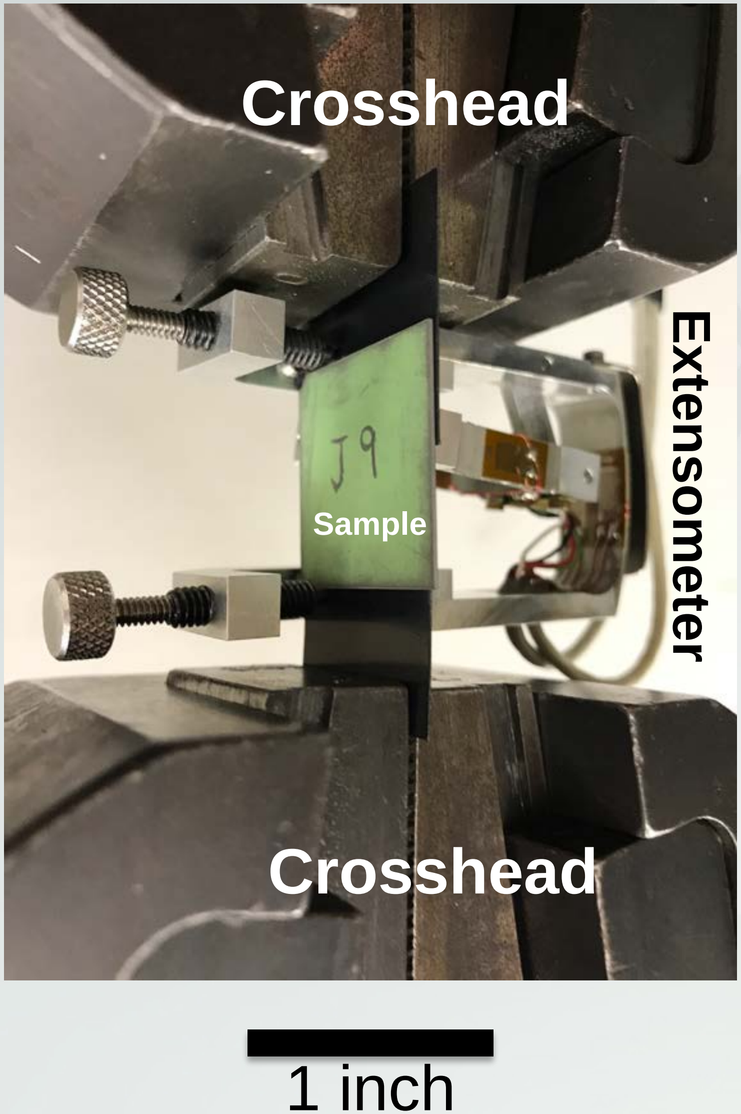
30 Mins Brazed Sample (After Oxidation)

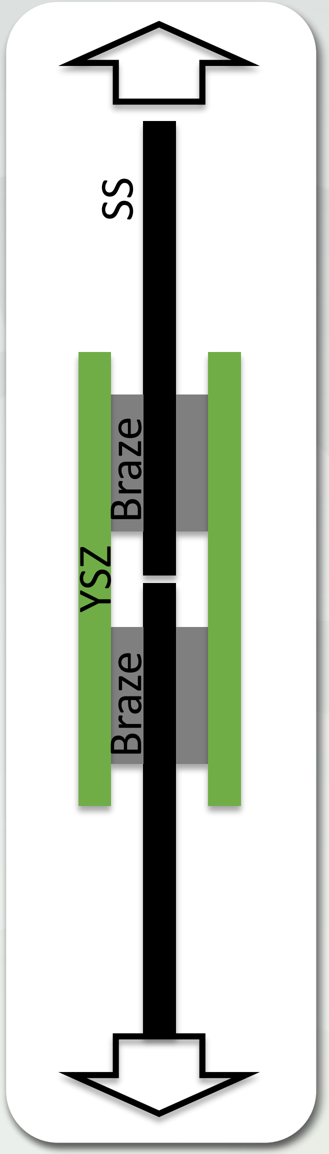
A Protective Chromia Scale Forms on the Stainless Steel after Oxidation





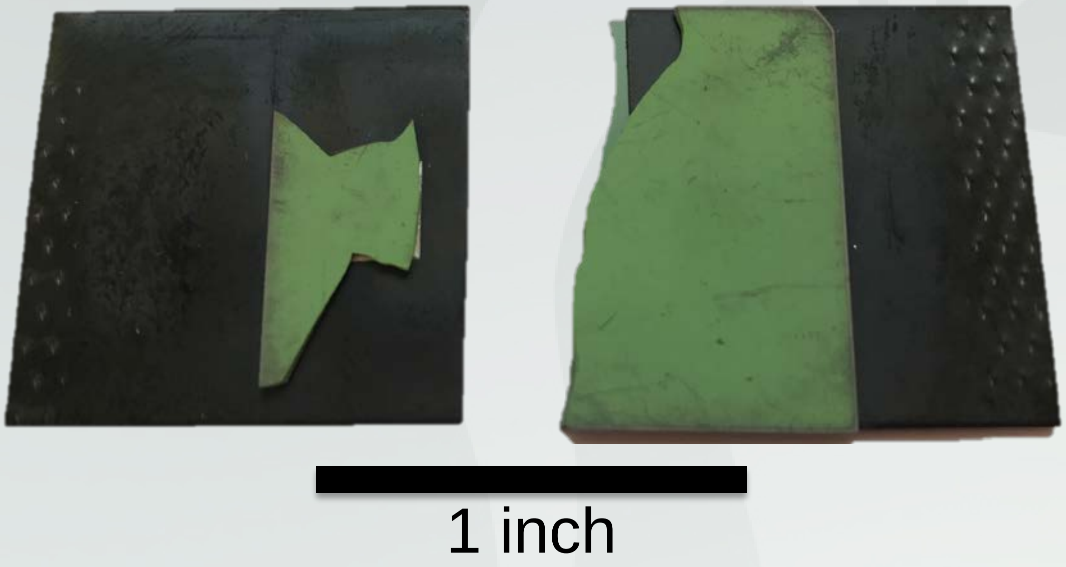
Symmetric Double Shear Lap Set-up with Tensile Loading





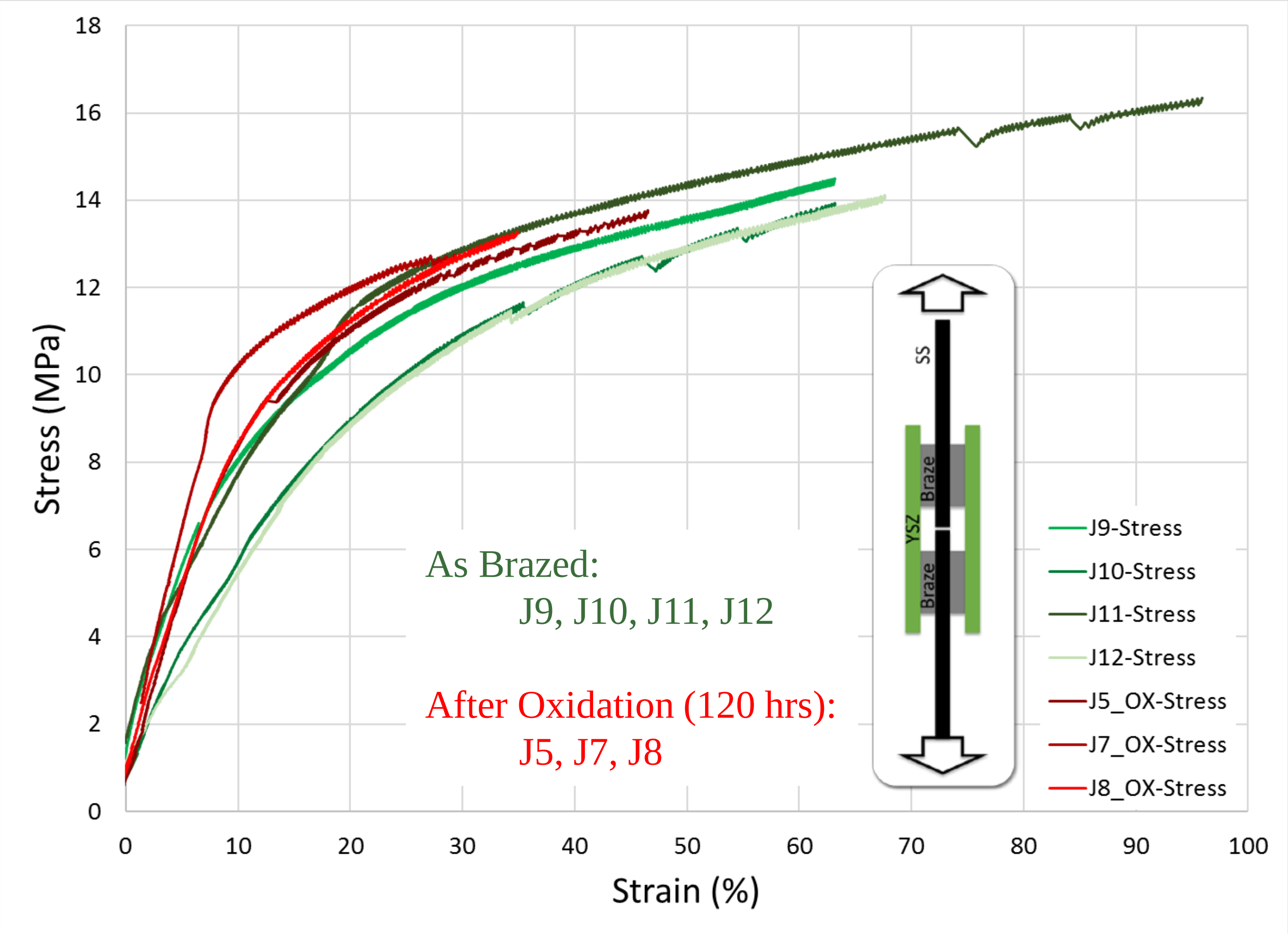
- Displacement Control: 0.009mm/min (~10^{-3} s^{-1})
- Test to Failure

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



- Almost 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.

Tests Show Good Ductility Followed by Brittle Fracture



δ: Displacement per joint; σ_{shear}: Shear stress; ε_{shear}: Shear strain

a: ¼'' (lateral length of the joints); F: Load; t: Joint thickness;

δ = Extension/2; $\sigma_{shear} = F/2[a*(a-\delta)]$; $\varepsilon_{shear} = (\delta/2)/t$.

** Displacement measured with extensometers.*

Outline

- **Background and Motivation**
  - Benefits of Silver-Copper Brazes
  - Problems with Silver-Copper Brazes
  - Proposed Strategy
- **Experiments**
- **Results and Discussion**
  - Partially Sintered Ni Layers
  - As Brazed Joint Microstructures
  - Oxidized Braze Joints
  - Mechanical Properties with/without Oxidation
- **Conclusions**

The Ag-Ni Braze Has Several Key Benefits

Pore Type	Reactive Air Brazing	Ag-Ni Brazing
Type I (wetting) Pore Formation	• ***θ = 45°*** (for Ag-4CuO) occasionally leads to pores during manufacturing [1,2]. • Organics in the braze paste can also lead to pores during manufacturing [3].	• θ = 30° 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 ~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.

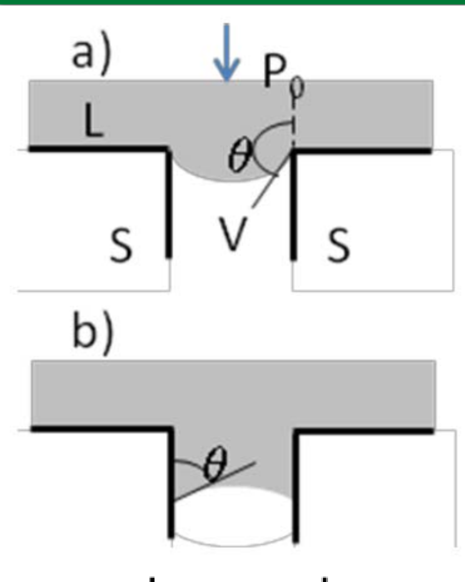
[1] Kim, J.Y. et. al, Journal of the American Ceramic Society, 2005.
[2] Kim, J.Y. et. al, Journal of Materials Research, 2005.
[3] Bobzin, K. et. al, Advanced Engineering Materials, 2014.
[4] Bause, T., et al., Fuel Cells, 2013.
[5] Trumble, K.P., Acta Materialia, 1998.
[6] Kim, J.Y. et. al, International Journal of Hydrogen Energy, 2007.

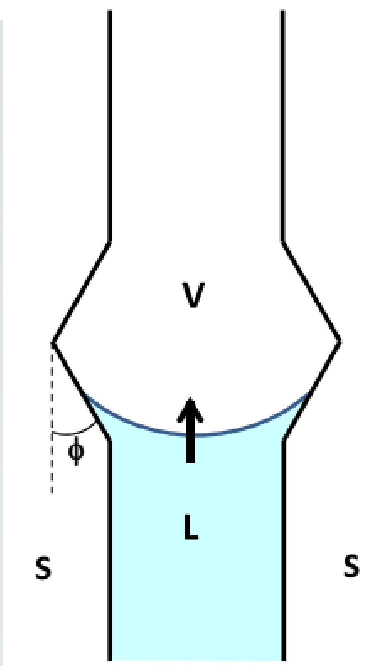
Conclusions

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 achieved.
4. The interfacial bonding strength with this new technique is stronger than the YSZ substrates after prolonged oxidation.
5. This technique may also be useful for enabling Ag-based (or other) brazes in other ceramic-ceramic and metal-ceramic applications.

Back up

Ni has good wettability to be infiltrated by Ag





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

$$h^2 = r_{eff}^2 \frac{\Delta P}{4\eta} t \quad \textit{(Washburn)}$$

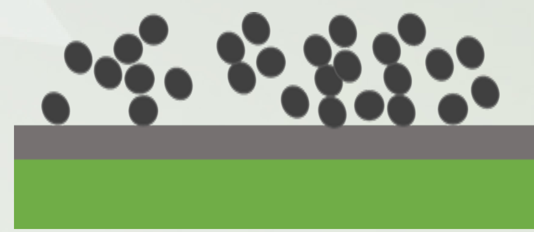
$$h^2 = r_{eff} \frac{\sigma_{LV}\cos\theta}{2\eta} t$$

h: Penetration depth;
θ: Contact angle;
r_{eff}: Effective pore radius;
P_C: Capillary pressure;
σ_{LV}: Surface tension;
ΔP: P_0-P_C, excess pressure;
η: Dynamic viscosity;
t: Time.

➔ *In equilibrium, infiltration happens when* $\theta < 90°$.

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* $\theta + \varphi < 90°$.

Eustathopoulos, N., Metals, 2015. ***5*** *(1): p. 350-370.*
Washburn, E.W., Physical Review, 1921. ***17*** *(3): p. 273-283.*

Ni has good wettability to be infiltrated by Ag

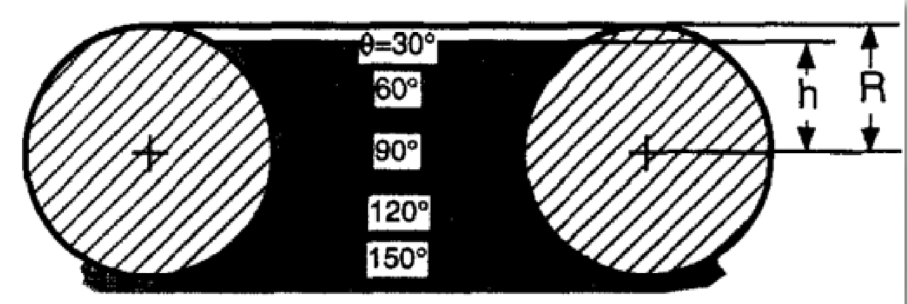
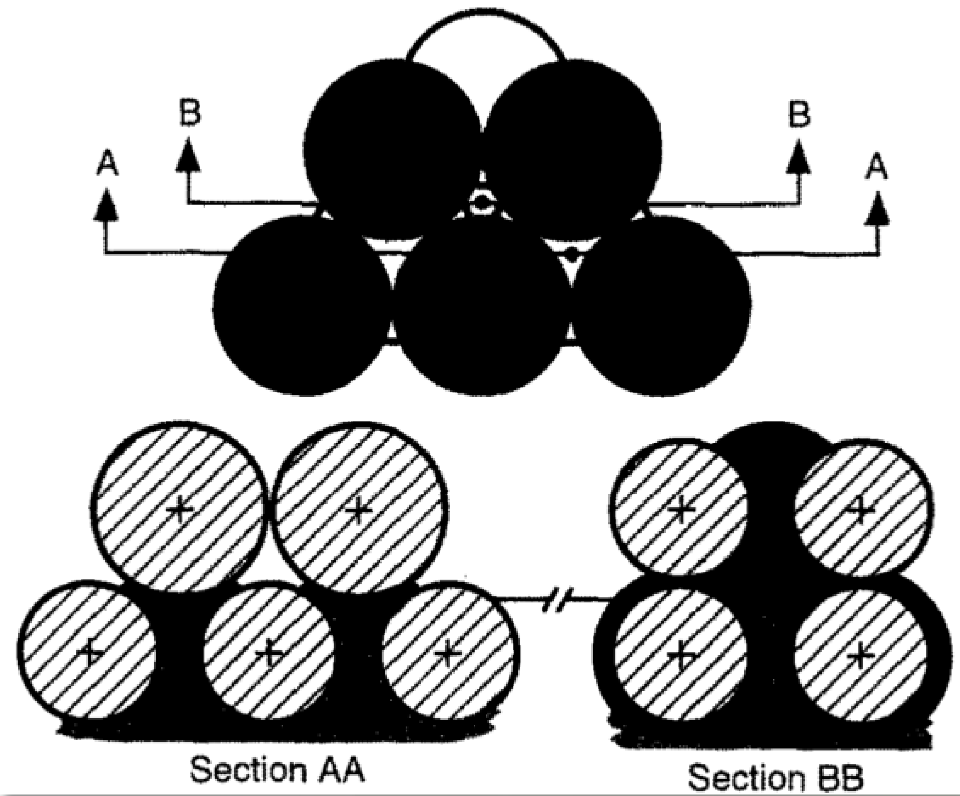
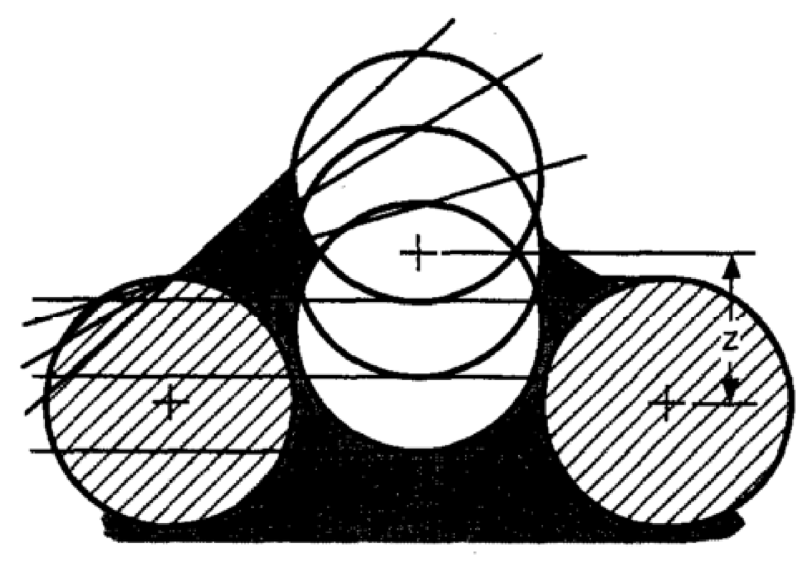


Fig. 4. Cross section of a toroid pore ($R/r = 1$) showing static liquid surface position as a function of contact angle for $\Delta P = 0$ (spontaneous infiltration).

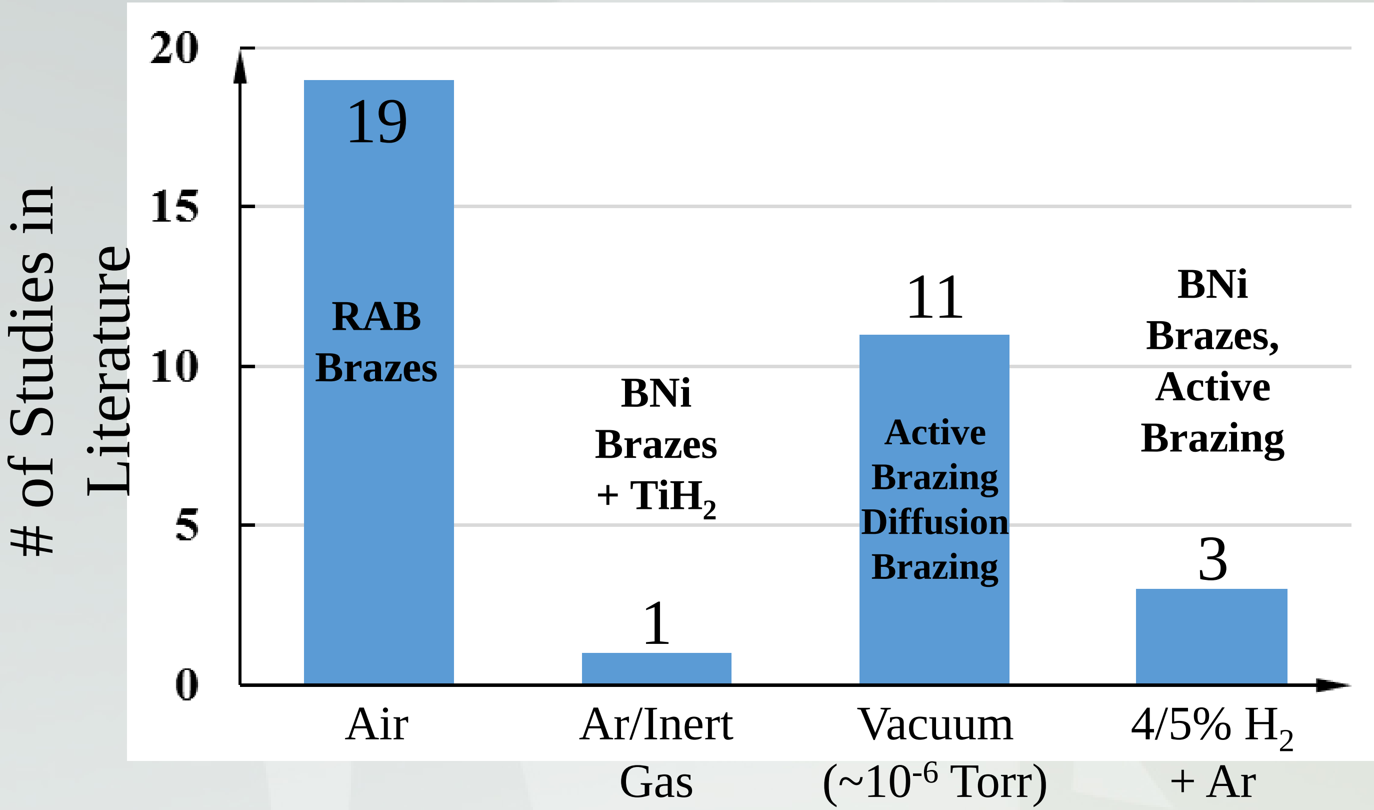




- Through calculation of the ratio between h and R critical wetting angles can be estimated based on packing geometry.
- Smallest critical wetting angle is **50.7°** for perfect penetration.

Trumble, K.P., *Spontaneous infiltration of non-cylindrical porosity: Close-packed spheres. Acta Materialia*, 1998. ***46*** *(7): p. 2363-2367.*

Brazing SOFC in Inert Gases is a Lightly Studied Topic



[1] Weil, K.S., Journal of Power Sources, 2005.
[2] Kuhn, B., Advanced Engineering Materials, 2014.
[3] Kim, J.Y., Journal of the Electrochemical Society, 2005.
[4] Weil, K.S., Electrochemical and Solid State Letters, 2005.
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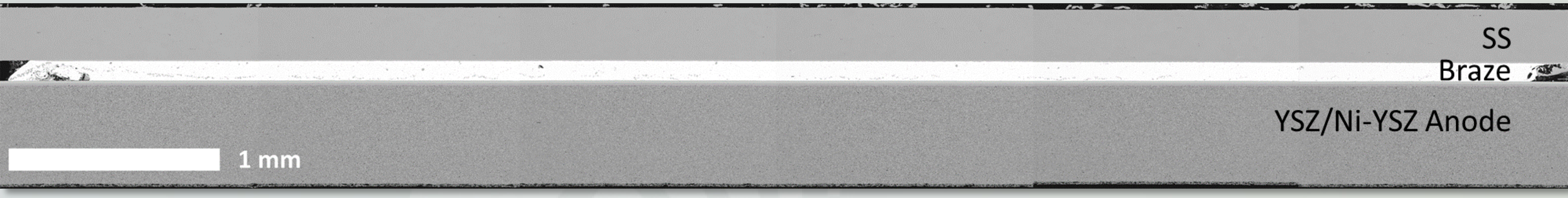
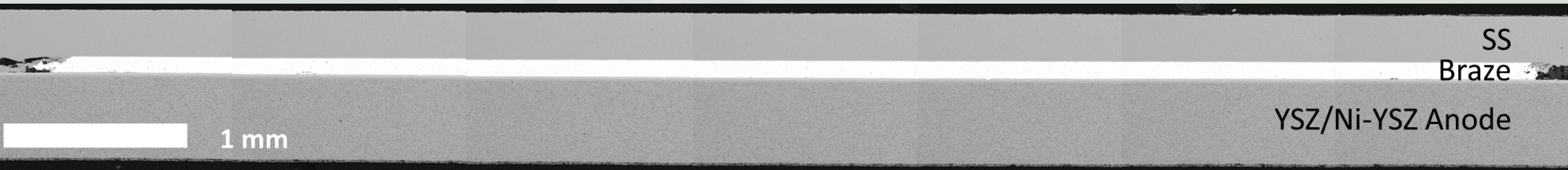
[33] Kim, J.-H., Journal of Fuel Cell Science and Technology, 2009.
[34] Lee, S., Journal of Alloys and Compounds, 2009.

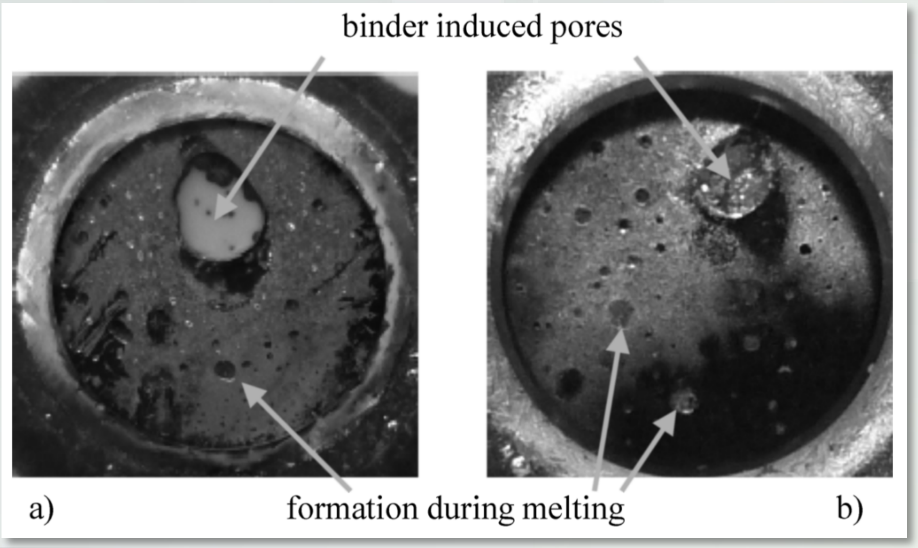
No Formation of Big Type I Pores with Ag Melt Impregnation

15 Mins Brazed Sample



30 Mins Brazed Sample

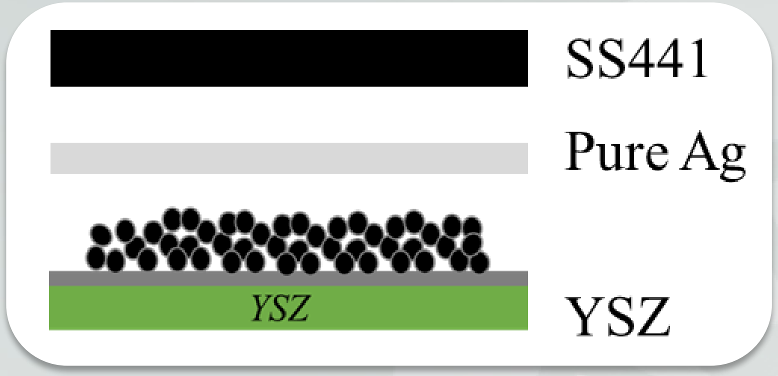


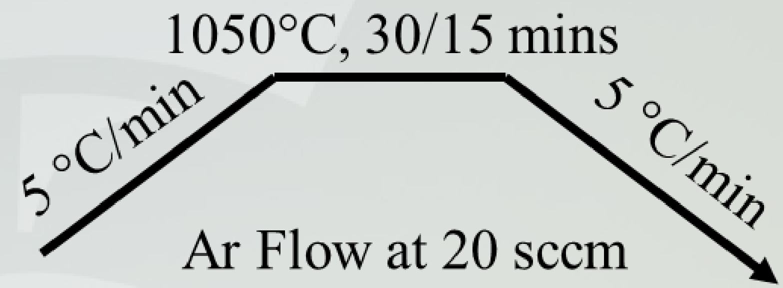


- Solid, dense joints were achieved with the Ag/Ni method using Ag foils.
- Whereas organics (binder) used in the paste form of braze filler materials often lead to big pores.

Bobzin, K., N. Kopp, and S. Wiesner, Influence of Filler and Base Material on the Pore Development during Reactive Air Brazing. Advanced Engineering Materials, 2014. ***16*** *(12): p. 1456-1461.*

Thickness of the Joints





Porous Ni interlayer: ~20 µms

+

Ag foil of 75 µms

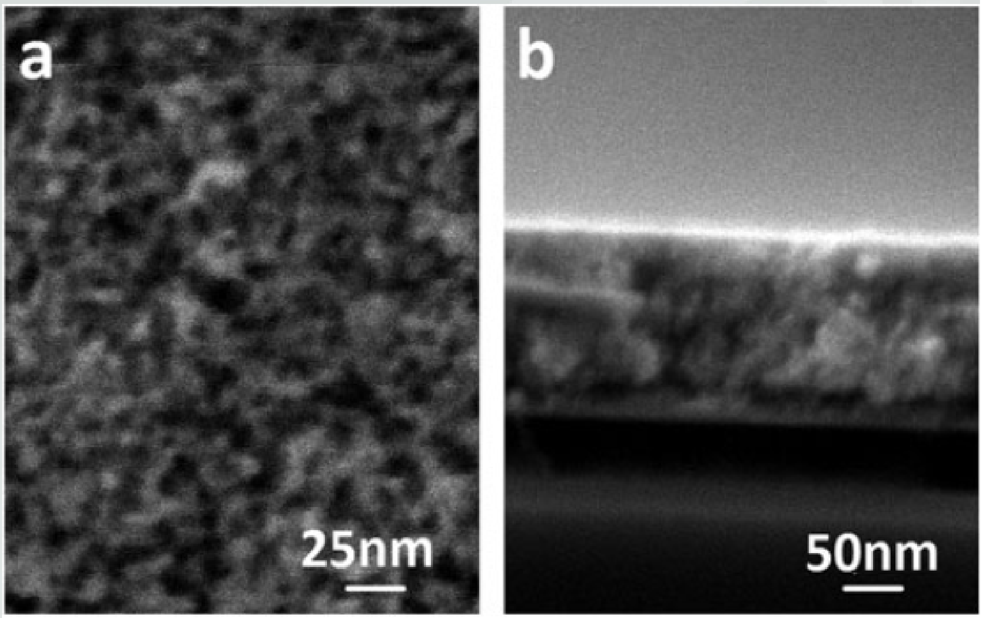
+

Some of the SS elements diffused into the braze

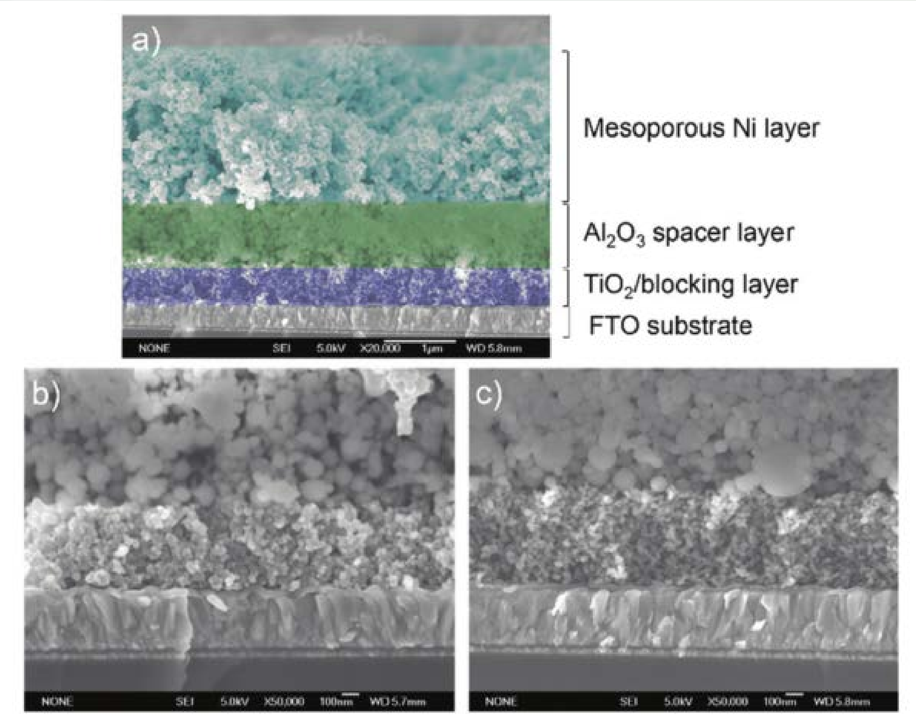
Joint thickness of ~100 µms

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 though chemical dealloying or simple reduction.



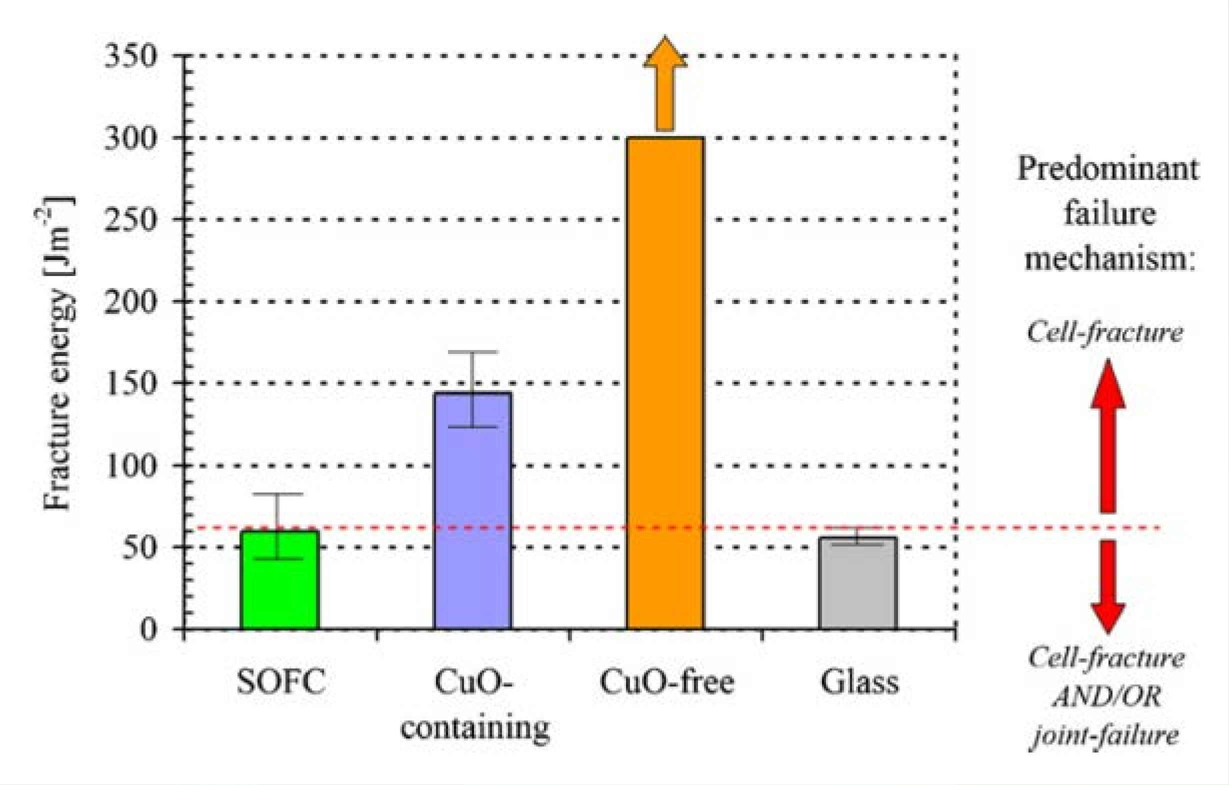
Chemically dealloyed $Ni_{33}Mg_{67}$ thin film (pure Ni)



Wang, L. and T.J. Balk, Synthesis of nanoporous nickel thin films from various precursors. Philosophical Magazine Letters, 2014. 94 (9): p. 573-581.

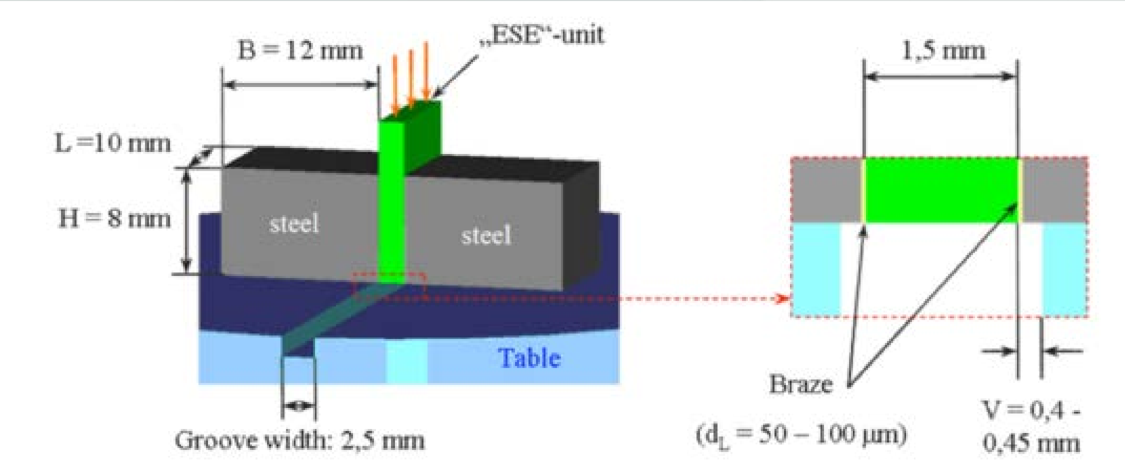
Ku, Z.L., et al., A mesoporous nickel counter electrode for printable and reusable perovskite solar cells. Nanoscale, 2015. 7 (32): p. 13363-13368.

On Ag joint strength

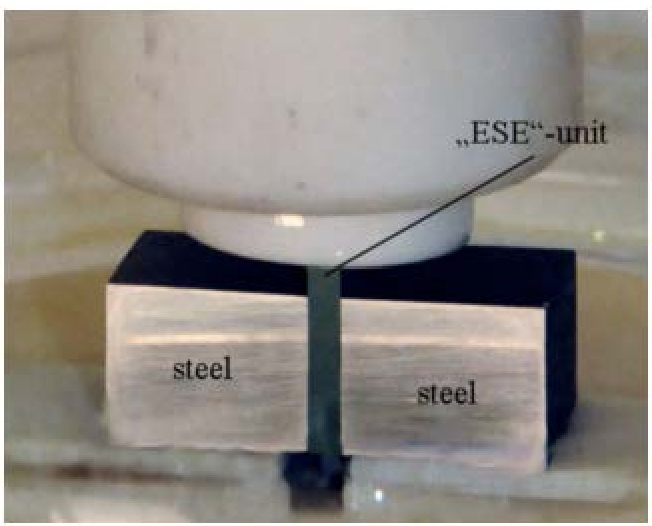


Ku, Z.L., et al., *A mesoporous nickel counter electrode for printable and reusable perovskite solar cells.* Nanoscale, 2015. **7** (32): p. 13363-13368.

Kuhn, 2014



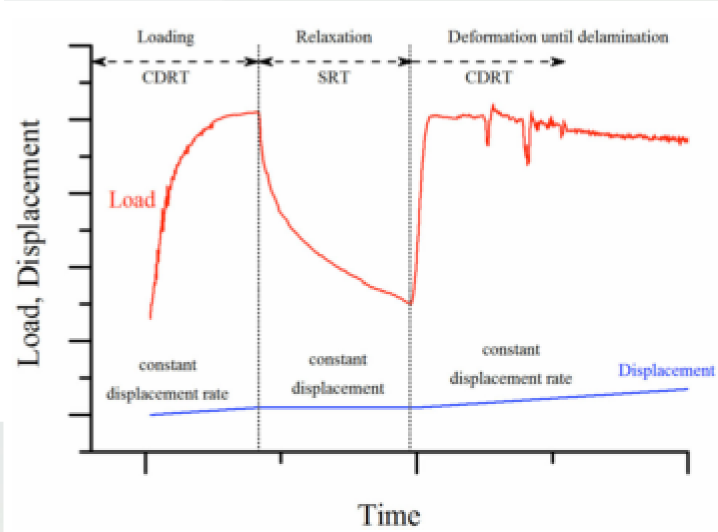
Ag foils of 100 μm thickness (produced by Schlenk GmbH, purity: 99.995%) were applied as a simplified standard RAB. The braze foils were cut, chamfered, and



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

While the total strain

$$\gamma_{tot.} = \frac{\delta_{corr.}}{d_L}$$



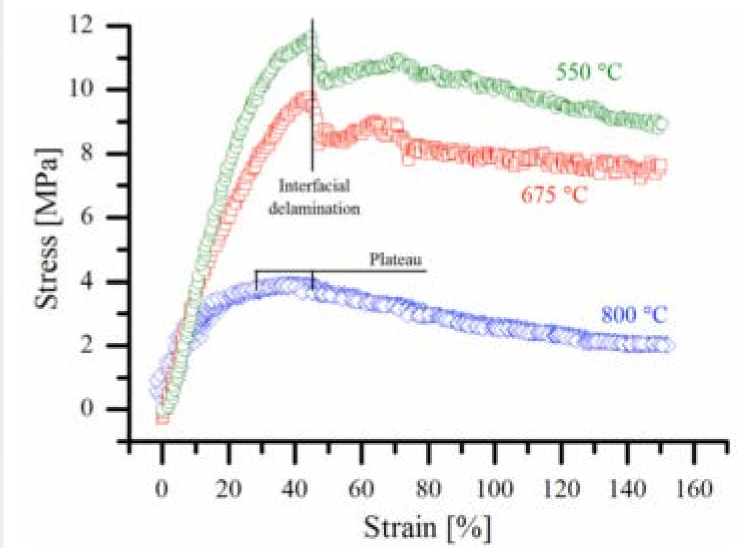
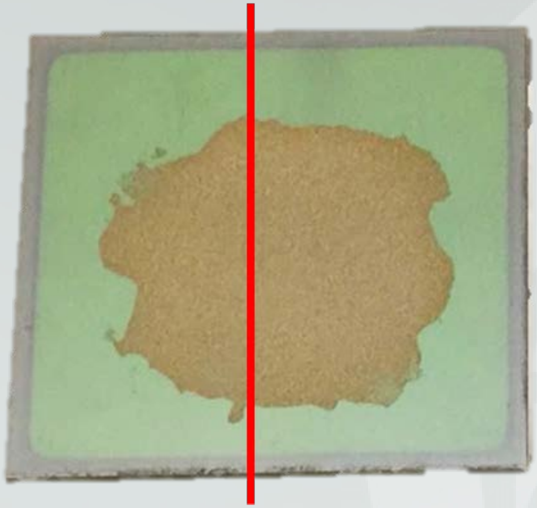
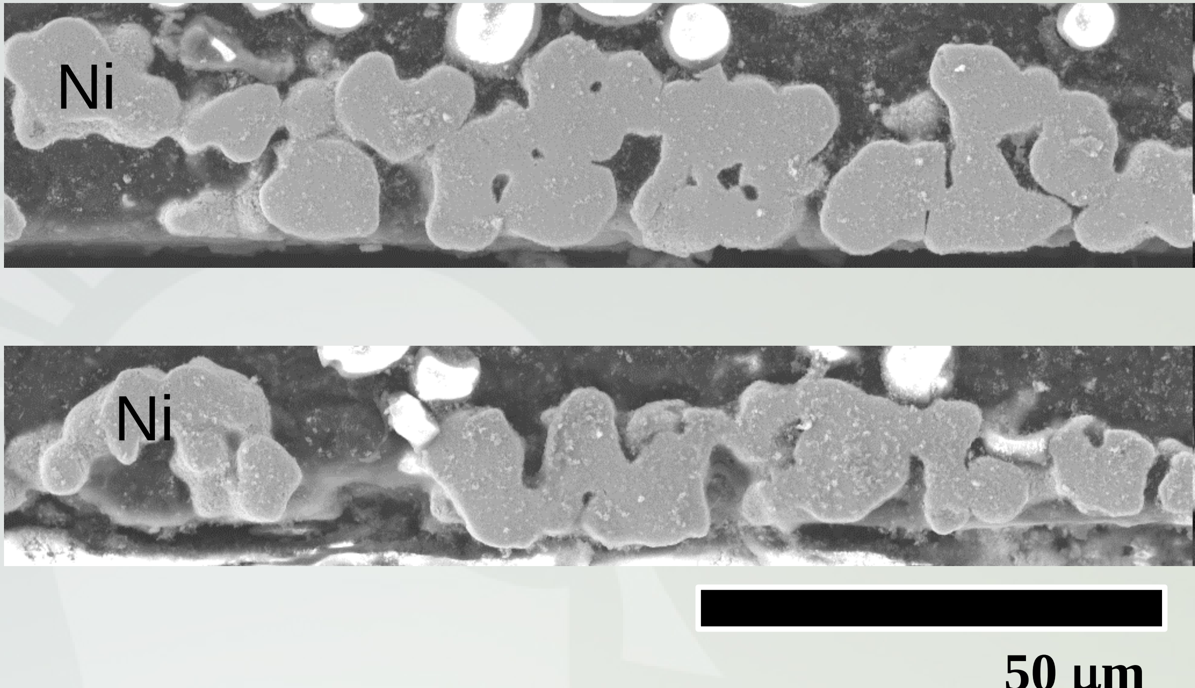


Fig. 5. Stress-Strain curves of as-brazed pure Ag-joints at 550, 675, and 800 °C in air.

Porous Interlayer of Ni Layer was Achieved on the YSZ



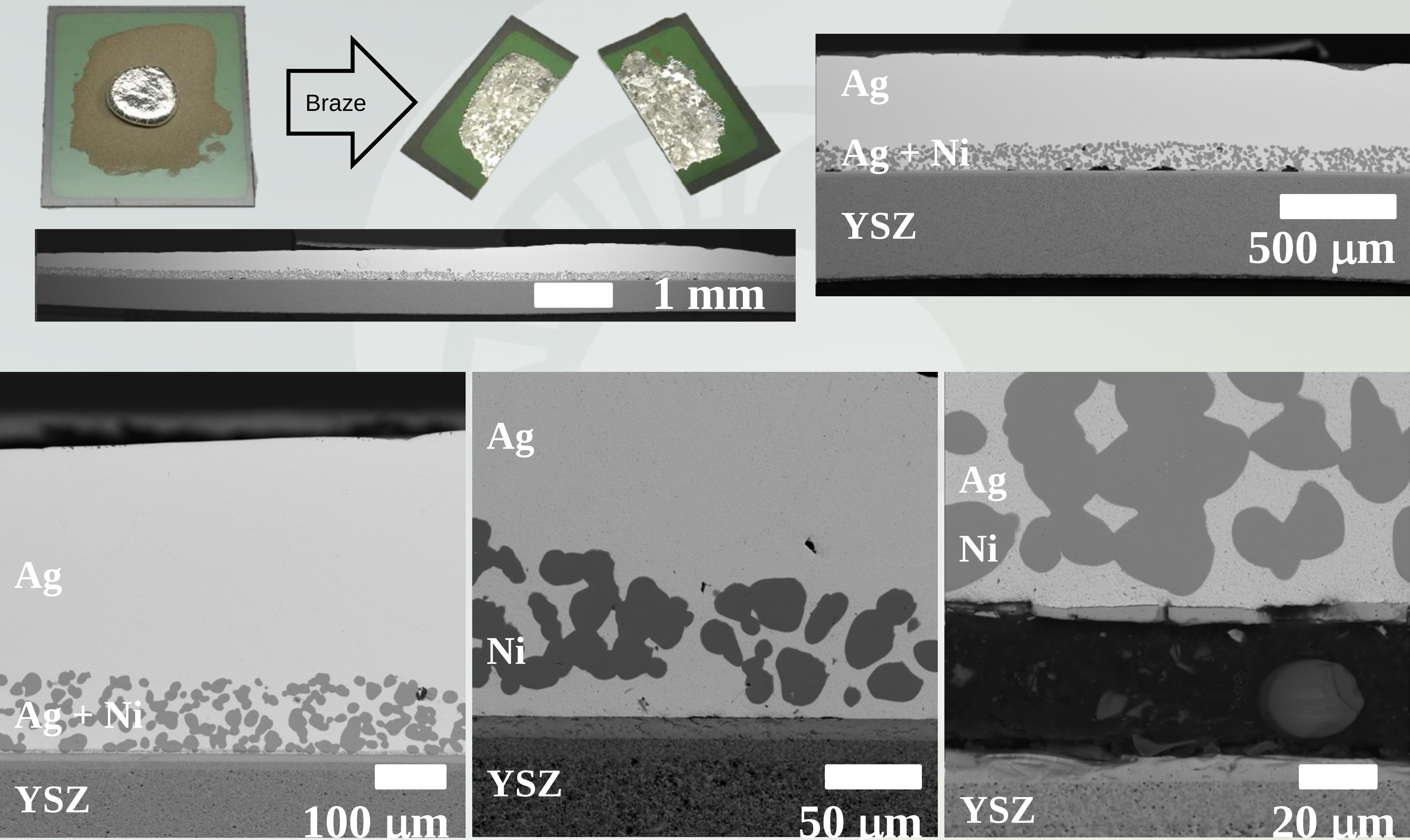


- Sample cross-sectioned to examine the as-sintered microstructure.

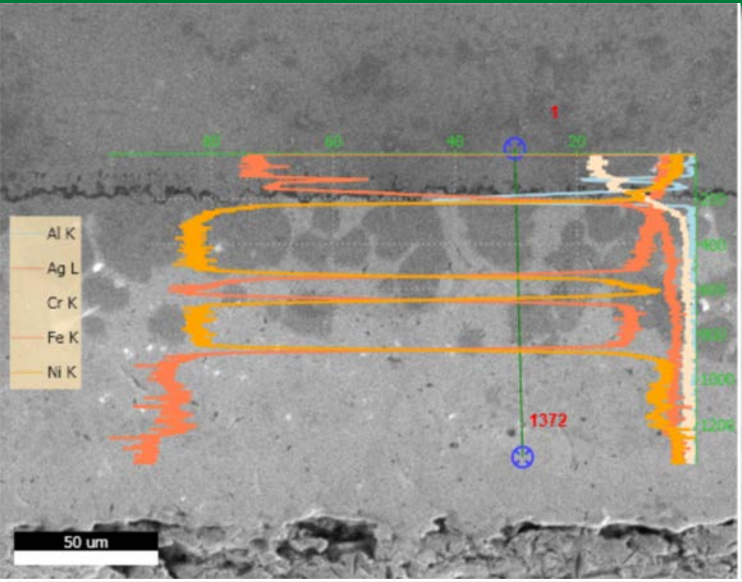
- Hot mounted @ ~30kN, ~190° C

A partially sintered Ni particle interlayer has formed on the YSZ surface.

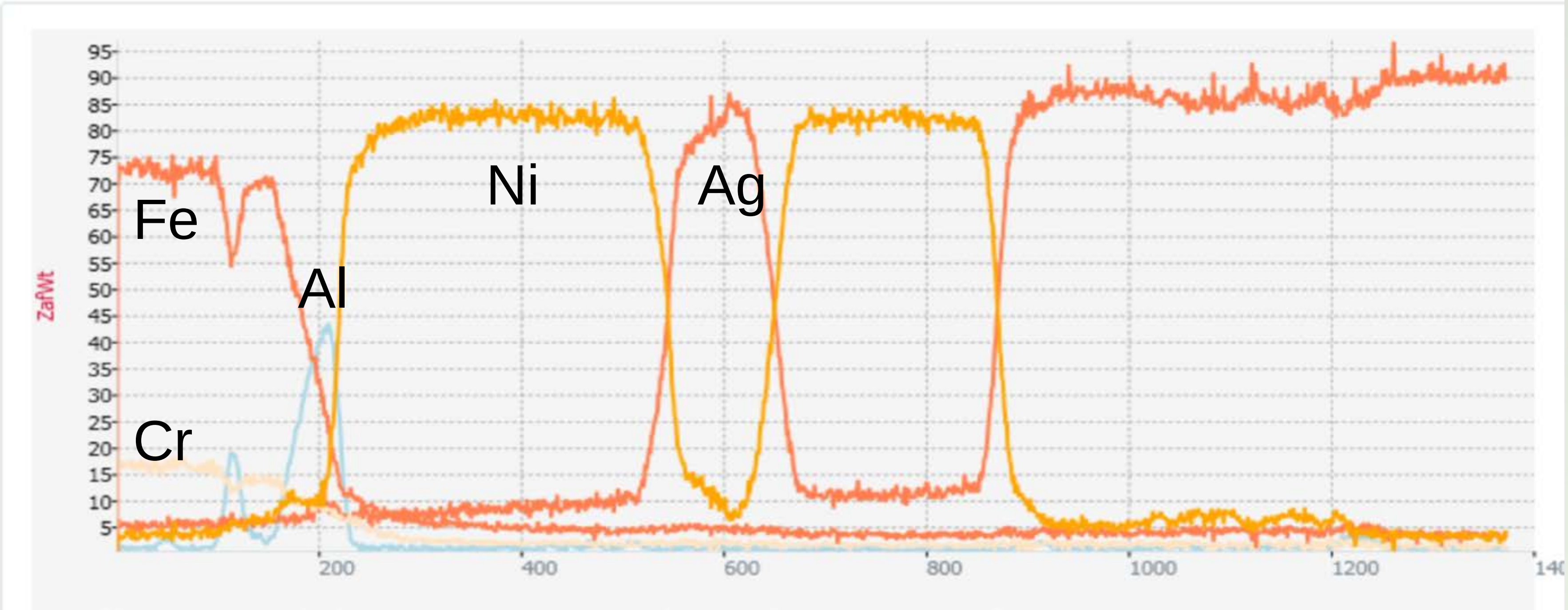
No formation of big Type I pores with Ag infiltration



On Al_2O_3 surface scale



The Al_2O_3 layer has very limited effect on Fe and Cr diffusion.



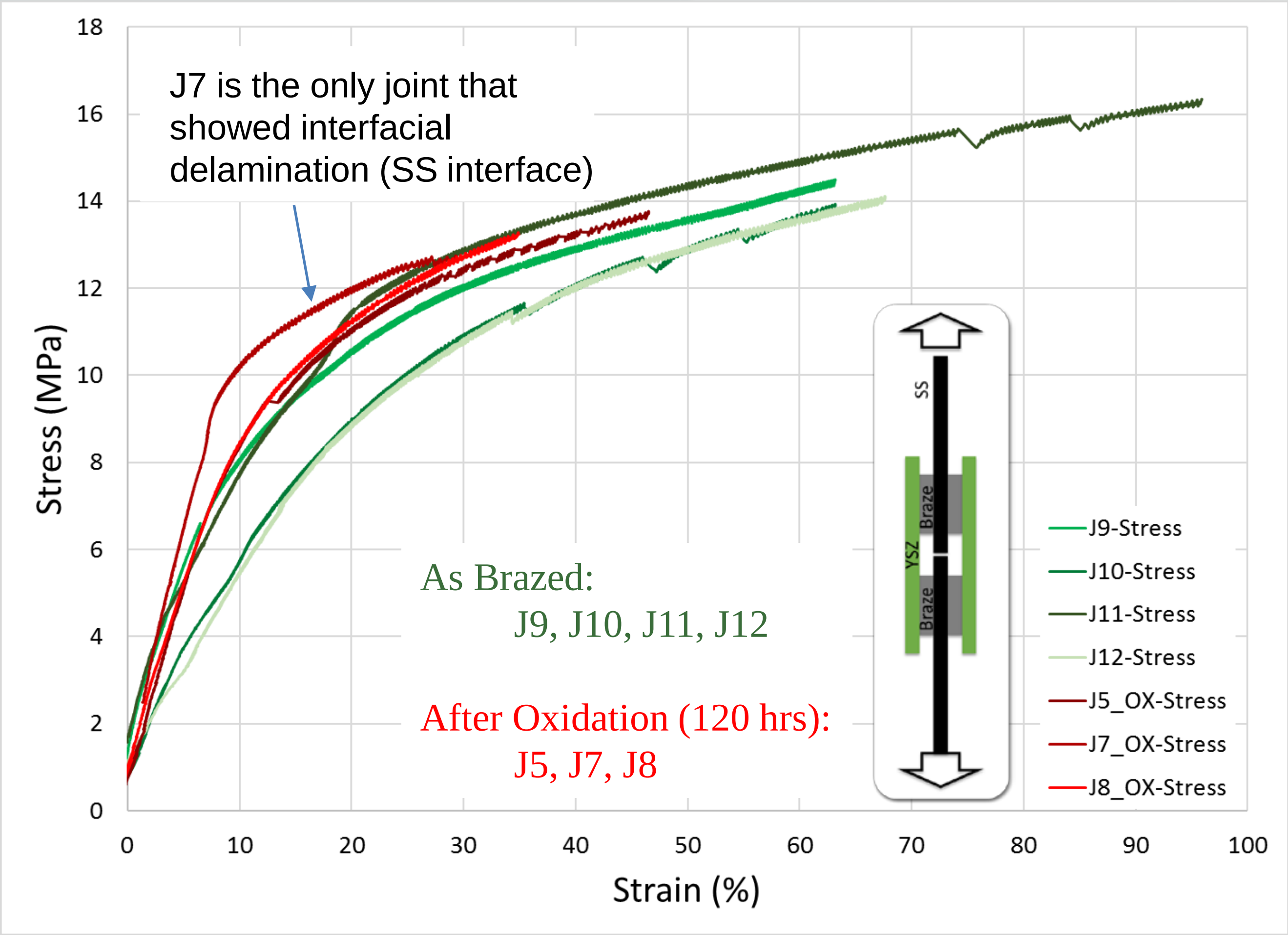
Ag-Ni Braze Optimization

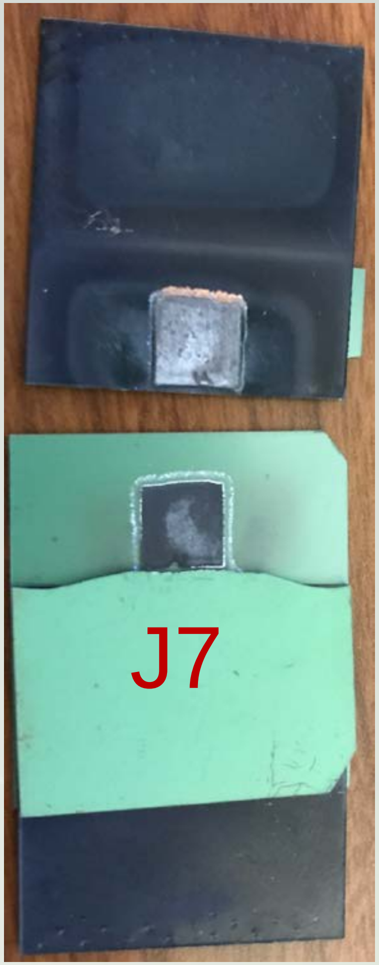
1. Different Ni particle sizes
    - The smallest particle size that can still provide good "wetting" of the Ag on YSZ;

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).

Only 1 out of 12 Joints Shows Delamination after Oxidation





- The crack probably occurred inside the oxidized reaction layer.

* *Displacement measured with extensometers.*

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

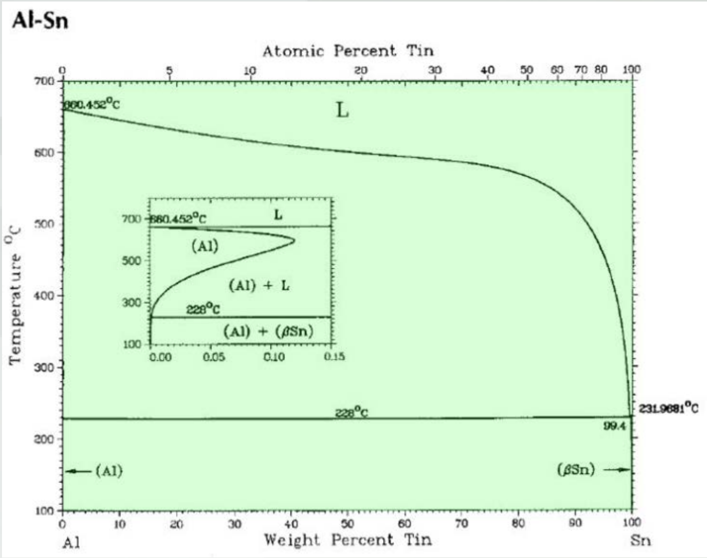
2. Test braze ***joint strengths*** (tensile tests done here at MSU)
   - Systematic tests to obtain statistically meaningful strength;
   - Bend tests to assess the bonding strength.

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

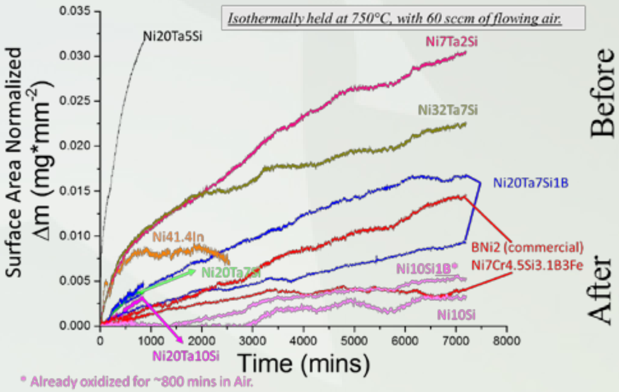
4. Test braze joint ***permeability*** (at MSU)
   - According the previous literature study, the metallic/braze seal are much better in gas permeability in TMC tests.
   - Also, consider the Type II pores to be suppressed in the long run, gas tightness in operation should be better.

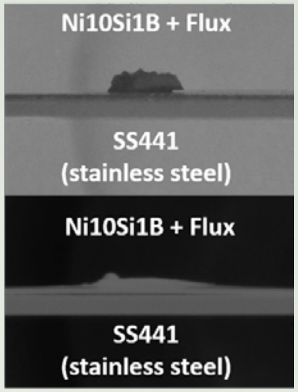
Porous Metal Interlayers for Other Brazing Systems

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.





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Likely Impact

1. The new Ag-Ni brazing system will be the go-to braze for SOFCs
   - No Type I or Type II pores;
   - Better ***ductility*** and ***bond strength*** compared to RAB brazes;
   - ***Cost effective*** production compare to active brazing techniques;
   - Possible longer operation time for SOFC devices.

2. ***Brazing with infiltration*** opens up new ways to braze dissimilar parts *in ceramic-metal joining and ceramic-ceramic joining.*

Future Publication Plan

1. "New Braze Materials and Techniques for Solid Oxides Fuel Cell Applications", ***Quan Zhou***, PhD Thesis, 2018.

2. "Transient Porous Metal Interlayers for Improved Metal-Ceramic Brazing", ***Quan Zhou***, Thomas R. Bieler, Jason D. Nicholas, Advanced Materials, 2017. (In Preparation)

3. "Dual Atmosphere Rapid Thermal Cycling Stability of Ag-Ni Brazes for Solid Oxide Fuel Cell Applications", ***Quan Zhou***, Rick Kerr, Thomas R. Bieler, Jason D. Nicholas, Journal of Power Sources, 2017. (In Preparation)

4. "Processing Effects on the Microstructure, Oxidation Resistance, and Performance of Silver Braze Solid Oxide Fuel Cell Joints Utilizing Porous Nickel Interlayers", ***Quan Zhou***, Thomas R. Bieler, Jason D. Nicholas, Journal of Materials Science, 2017. (In Preparation)

5. "Experimental Evaluation of Computationally Identified Nickel-Based Brazes for Stainless Steel to Yttria Stablized Zirconia Joining", ***Quan Zhou***, Tridip Das, Yuxi Ma, Joe Phongpreecha, Thomas R. Bieler, Yue Qi, Jason D. Nicholas, Journal of Alloys and Compounds, 2018. (In Preparation)

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