4-17-2019

Characterization and Failure Analysis of Solid-State Diffusion Bonded Ceramic-to-Metal Transitions

Yaiza Rodriguez
Boise State University

Timothy L. Phero
Boise State University

Luke Schoensee
Boise State University

Allyssa Bateman
Boise State University

Kyu Bum Han
HiFunda, LLC

See next page for additional authors

Name
Yaiza Rodriguez, Timothy L. Phero, Luke Schoensee, Allyssa Bateman, Kyu Bum Han, Jim Steppan, Balakrishnan Nair, and Brian J. Jaques

Characterization and Failure Analysis of Solid-State Diffusion Bonded Ceramic-to-Metal Transitions

BOISE STATE UNIVERSITY
COLLEGE OF ENGINEERING
Micron School of Materials Science and Engineering

Yaiza Rodriguez[1], Timothy L. Phero[1], Luke Schoensee[1], Allyssa Bateman[1], Kyu Bum Han[2], Jim Steppan[2], Balakrishnan Nair[2], and Brian J. Jaques[1,*]

[1] Micron School of Materials Science and Engineering, Boise State University, [2] HiFunda, LLC

ADVANCED MATERIALS LABORATORY
MATERIALS FOR EXTREME ENVIRONMENTS AT BOISE STATE UNIVERSITY

I. Introduction & Background

Motivation

Heat exchangers that are operable in harsh environments (i.e., high temperature, high pressure) enable higher thermal efficiency in advanced power generation systems that use power cycles based on steam, helium (He) or supercritical CO_2 (sCO_2) (**Figure 1**).

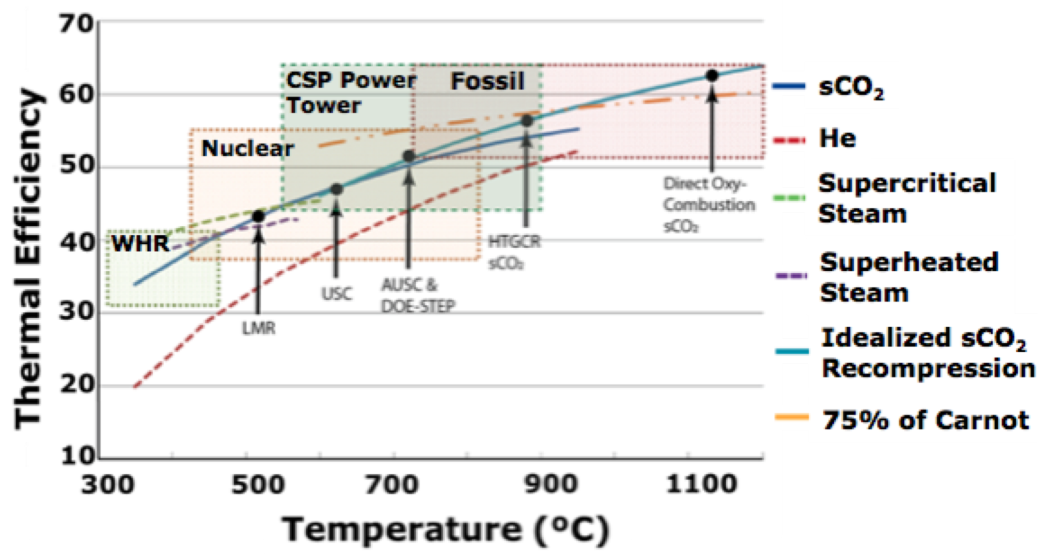


Figure 1: Thermal efficiencies at elevated temperatures for various turbine power generation systems [1]

At high temperatures (i.e., 800 °C) [2]:

1. Metals have a limited corrosion and creep resistance
2. Ceramics have a high corrosion and creep resistance

Solution

Joining ceramic heat exchangers to metal components exploits the higher operating temperatures of ceramics while utilizing an external metallic structure to increase the reliability.

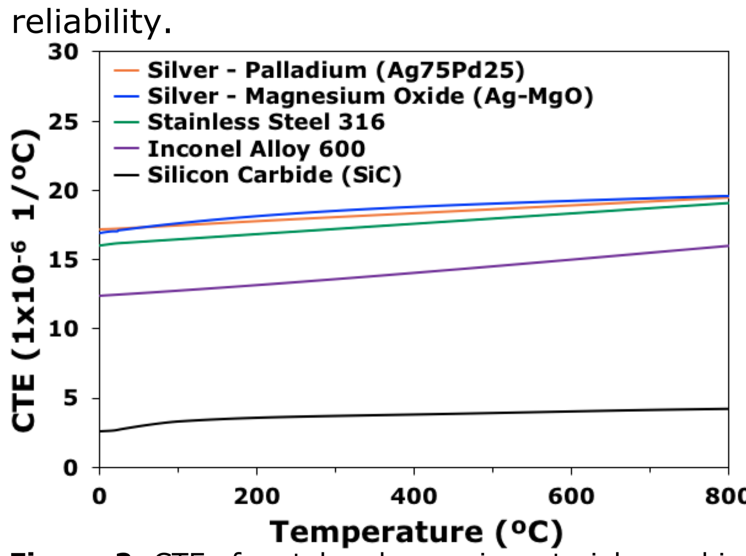


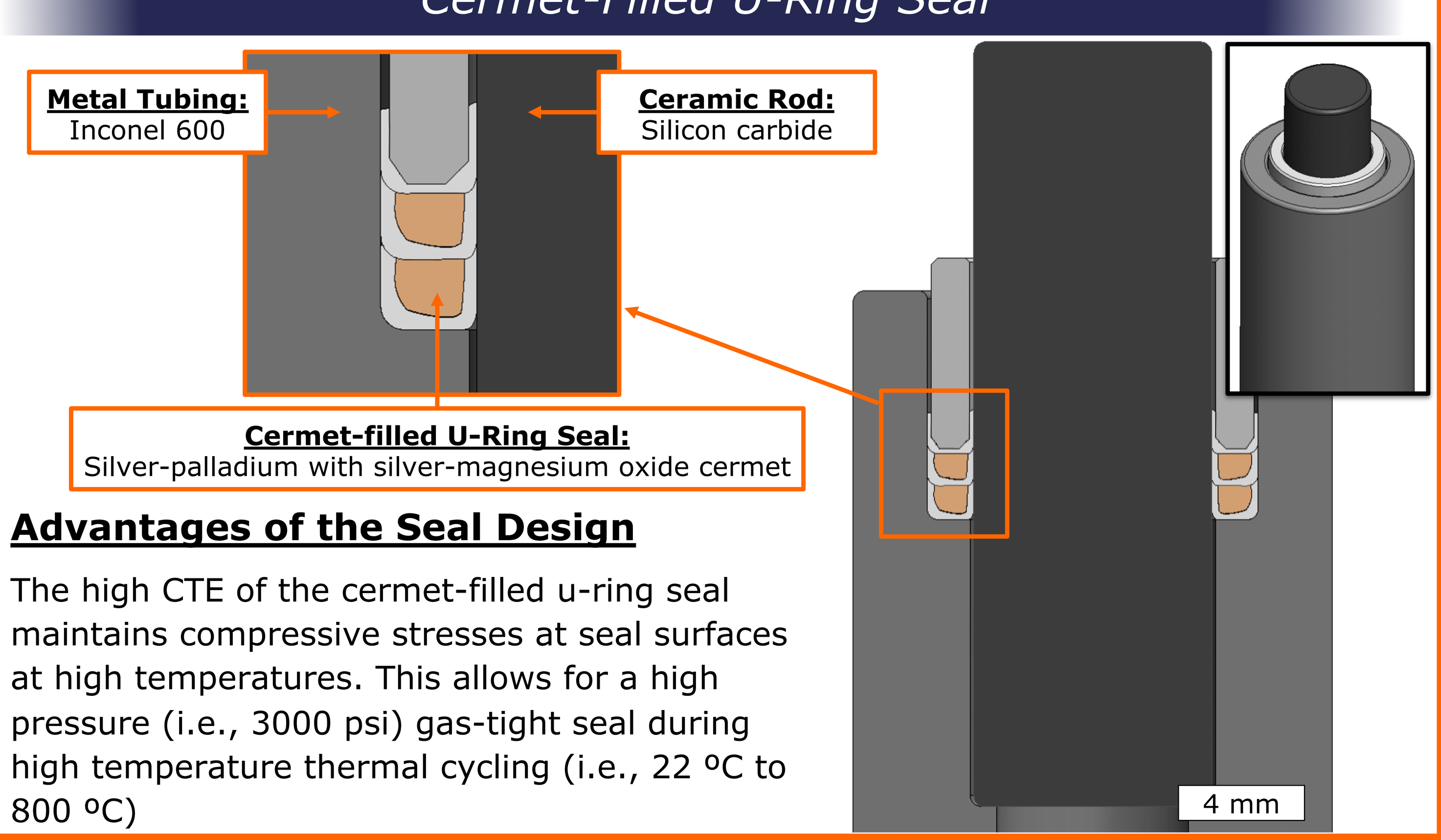
Figure 2: CTE of metal and ceramic materials used in our U-ring seal assembly samples.

Challenges

1. Mismatch in coefficient of thermal expansion (CTE) in ceramics and metals (**Figure 2**) induce stresses
2. Interactions between materials produce brittle phases
3. Variance in the manufacturing and processing produce differences in the mechanical integrity

II. Methods

Cermet-Filled U-Ring Seal



Advantages of the Seal Design

The high CTE of the cermet-filled u-ring seal maintains compressive stresses at seal surfaces at high temperatures. This allows for a high pressure (i.e., 3000 psi) gas-tight seal during high temperature thermal cycling (i.e., 22 °C to 800 °C)

III. Results: Characterization

Diffusion Bonding Disk

Characterization results from the diffusion bonding experiments on small disk samples (**Figure 3**), serve as a baseline to understand the effects of the forming parameters (i.e., temperature, pressure, time, geometry, cermet loading) as well as material interactions (i.e., chemical reactions, intermetallic phase formation, diffusion) in the joint samples.

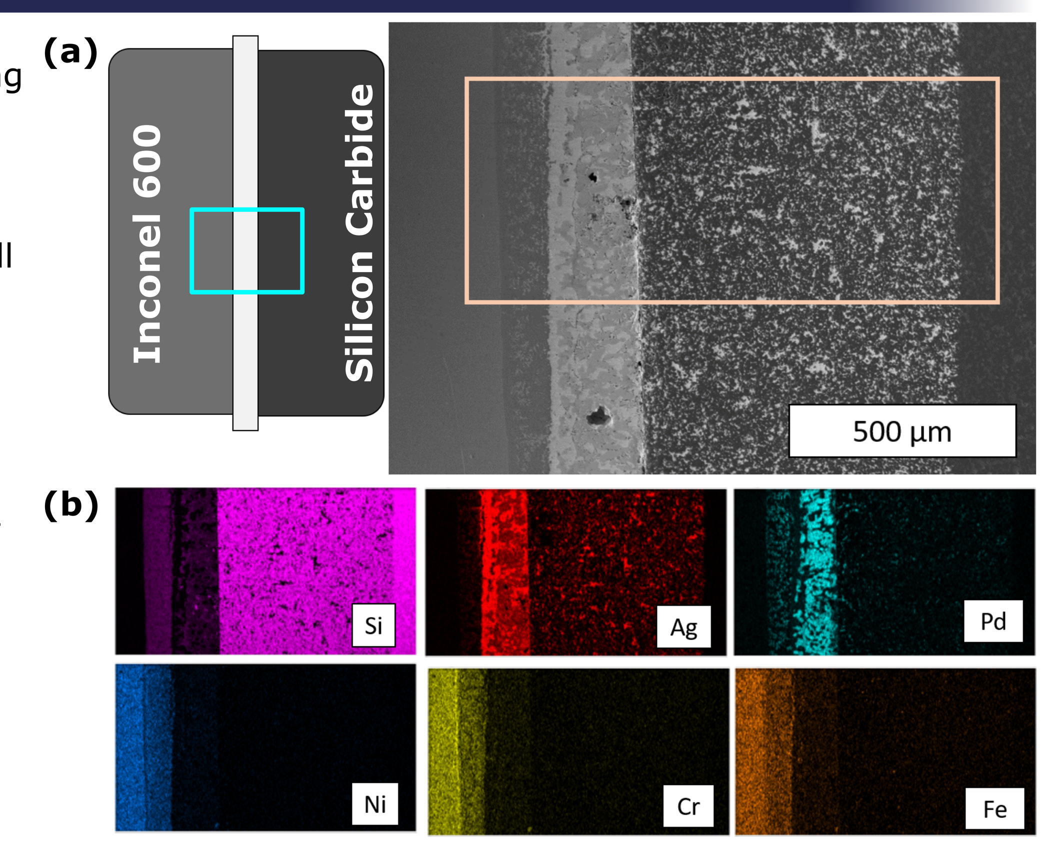
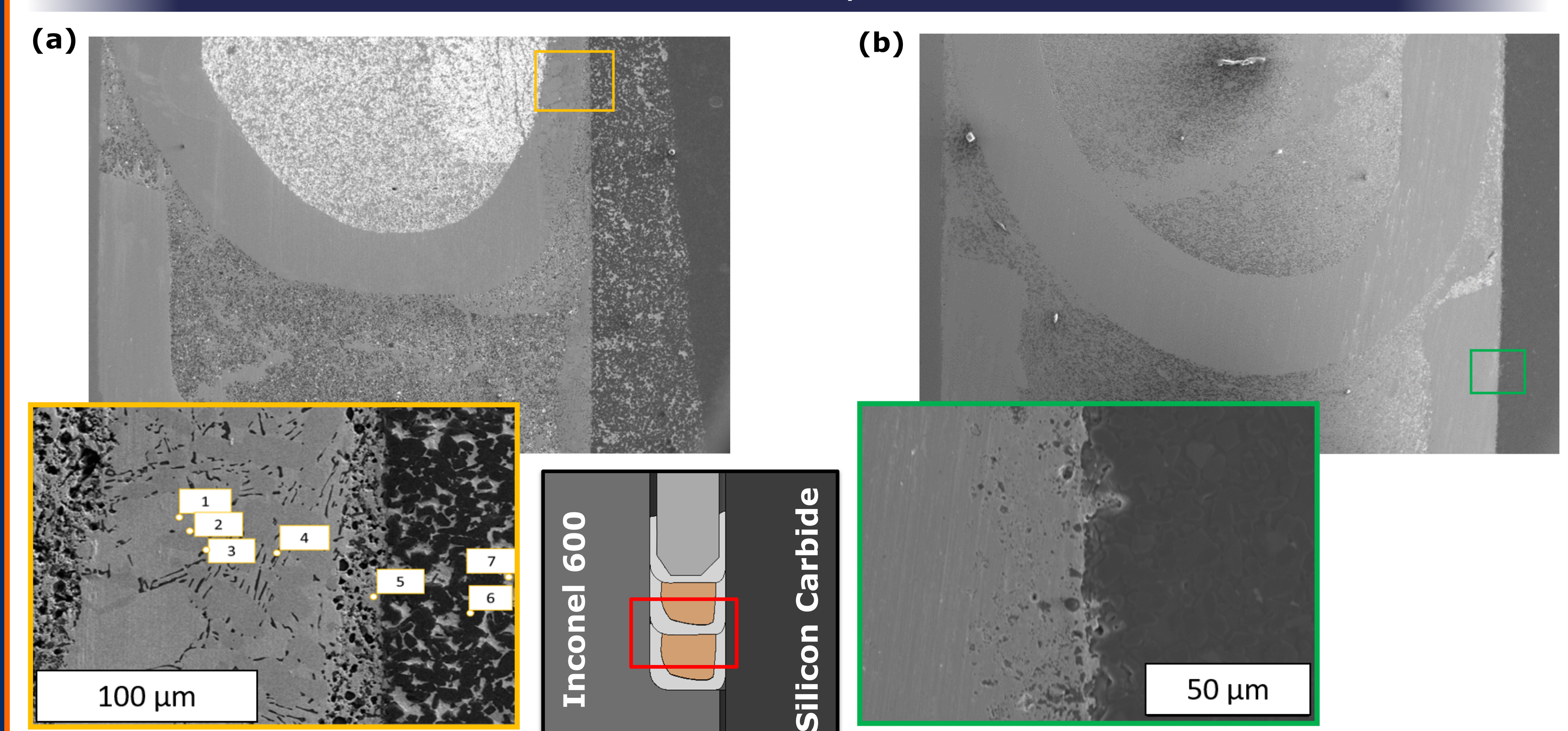


Figure 3: (a) Schematic representation and scanning electron microscopy (SEM) cross-section of Inconel 600/Ag75Pd25/SiC joint formed at 930 °C, and 1 MPa, for 180 minutes showing diffusion of free Si into the metal side as well as diffusion of Ag and Pd into the ceramic side. (b) Energy dispersive x-ray spectroscopy (EDS) map scans of joint shown in where phase separation of Ag and Pd from the AgPd interlayer can be observed at the interface. In addition, formation of intermetallic-silicide phases due to the diffusion of free Si from SiC into the metal side was discovered.

Full Assembly Joint



(c)

Point	C	O	Mg	Si	Pd	Ag
1	21	4	0	0	0	74
2	33	2	0	21	41	3
3	18	1	0	76	2	4
4	16	0	0	78	0	6
5	27	7	0	4	7	55
6	54	0	0	46	0	0
7	20	5	0	1	0	73

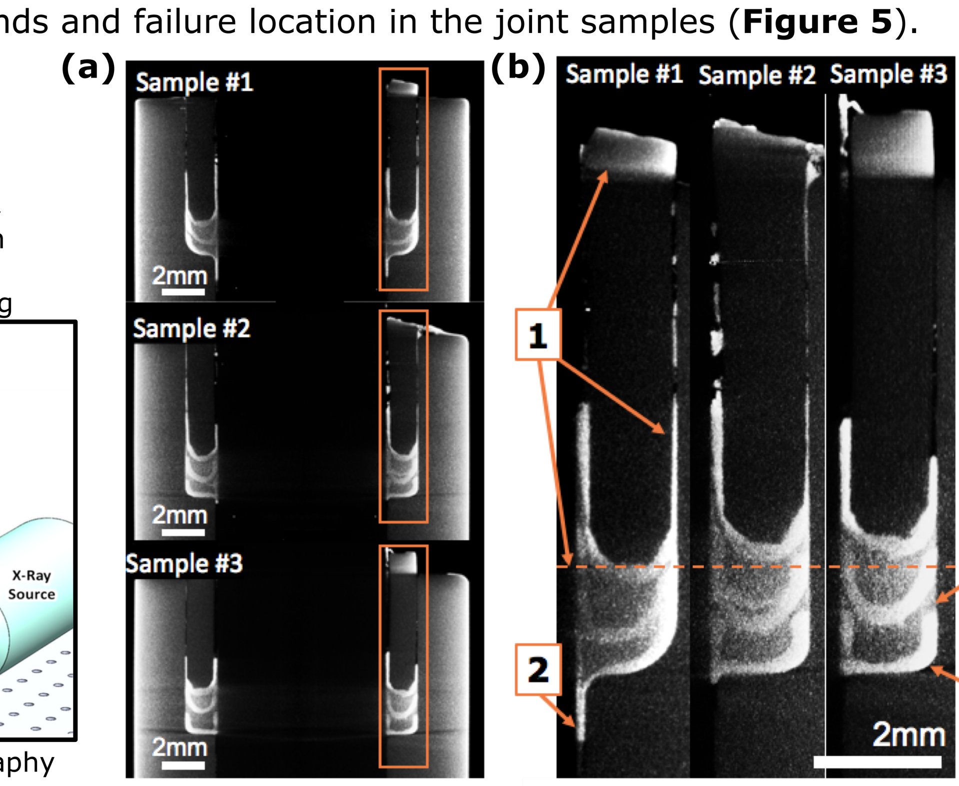
Figure 4: SEM cross-section images of joint samples (a) Inconel 600/Ag75Pd25/SiC with AgPd-MgO(cermet), and (b) Inconel 600/Ag75Pd25/SiC with Ag-MgO(cermet). Both assemblies were formed at 940 °C, and 104 MPa, for 180 minutes. (a) Assembly was heated/cooled at 3 °C/min whereas (b) was heated/cooled at 10 °C/min. Diffusion of Ag and Pd from the U-ring into the SiC increases as the heating/cooling rate decreases. Solid-state reactions increase with a lower heating/cooling rate introducing new phases. Also, it appears that the addition of Pd in the cermet (a) contributes to the increase in diffusion. (c) Table of EDS point scans, atomic %, shows the Ag/Pd phase separation, and the possible phase formations due to Pd and free Si interactions at the U-ring/SiC interface.

IV. Results: Failure Analysis

X-ray Computed Tomography

X-ray computed tomography (XRCT) (**Figure 6**) was used as a non-destructive, non-invasive technique for visualizing general trends and failure location in the joint samples (**Figure 5**).

Figure 5: (Right) (a) XRCT renderings of three seal samples. (b) Close-up of U-ring and Top-hat positioning on the right-hand side; the following observations lead to modeling efforts – **1:** Top hat deformation, **2:** U-ring plastic deformation, **3:** Leak path from excess cermet between U-ring, **4:** Variation in ledge radius on metal tubing



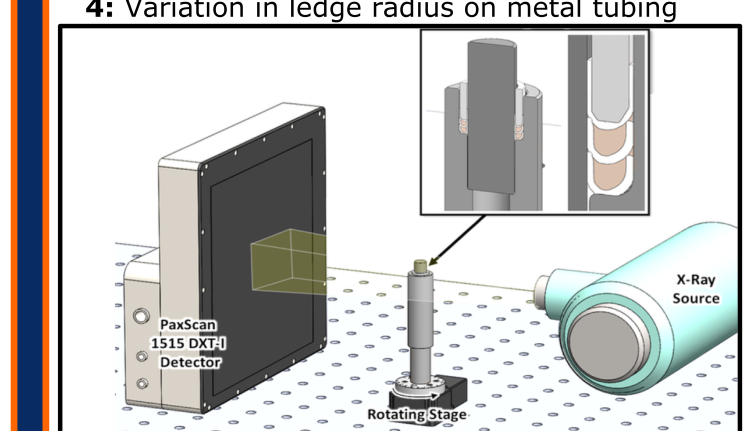


Figure 6: (Above) X-ray computed tomography (XRCT) schematic

Modeling – Finite Element Analysis

Variation in the machining and assembly of sealing components are detrimental to the sealing performance of the joint samples, as shown in XRCT (**Figure 5**). Computational modeling using finite element analysis (FEA) was used to:

1. Show the importance of machining tolerance (**Figure 7**) of the top-hat during the forming process
2. Optimize the dimensions of the sealing components by maximizing the contact force at the U-ring/SiC and U-ring/Inconel 600 tubing contact interfaces (**Figure 8**)

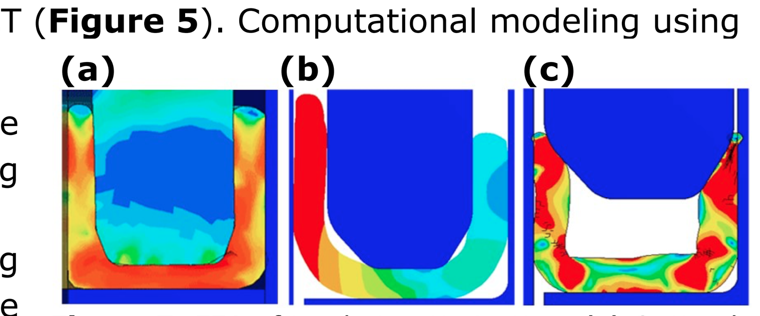


Figure 7: FEA of top hat optimization. (a) Optimal top hat width and placement. (b) Top hat is too narrow and off-center. (c) Top hat is too wide and placed too far to the right.

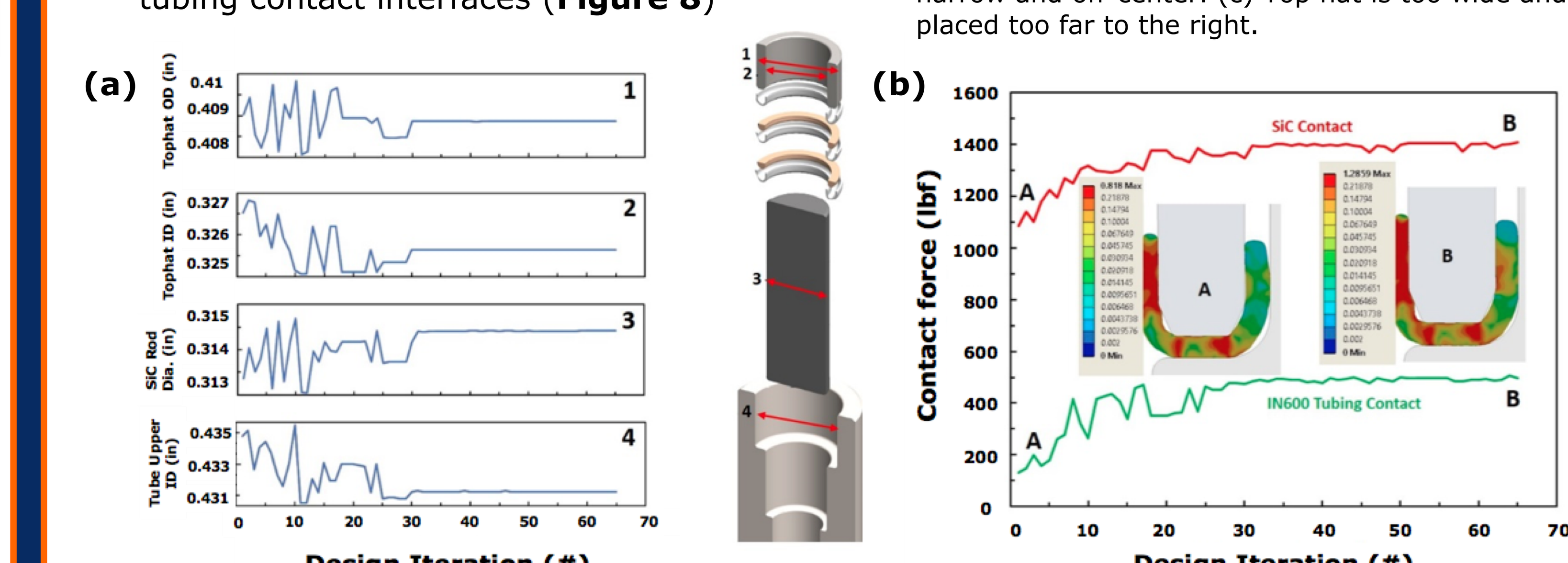


Figure 8: FEA shows that seal assembly dimensions (a) affect the contact force at the U-ring/IN600 and U-ring/SiC contact interfaces (b). Optimized dimension of the seal assembly was achieved by the maximizing contact force (b)

V. Conclusion

1. Diffusion studies and characterization of joints provided a better perspective and understanding about what to expect for future U-ring assemblies since temperature had the greatest effect on the diffusion behavior and intermetallic phases formation.
2. From XRCT and FEA, general trends were determined in the manufacturing and forming processes of the seal assemblies.

Contact

yaizarodriguezortego@u.boisestate.edu[1]
timothyphero@u.boisestate.edu[1]
brianjaques@boisestate.edu[1,*]

Micron School of Materials Science and Engineering
Boise State University, Boise, ID

References

[1] S. A. Wright, et al., "Workshop on New Cross-cutting Technologies for Nuclear Power Plants," in *Supercritical CO_2 cycle for advanced NPPs*, S. T. Inc., Cambridge, MA: MIT, 2017

[2] Lewinsohn, Charles A., et al., "Fabrication and joining of ceramic compact heat exchangers for process integration." International Journal of Applied Ceramic Technology 9, no. 4 (2012): 700-711.

Acknowledgements

This research is based upon work supported by the Department of Energy under SBIR Award Number DE-SC0015118.

Thank you to Richard Skifton at Idaho National Labs for the XRCT support.

hifunda
Innovative Energy & Materials Solutions

U.S. DEPARTMENT OF ENERGY

Idaho National Laboratory