

Welding of ZrB₂-Based Ultra-High Temperature Ceramics

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 - **Old welding system at MO-SCI for filler studies**
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Project Overview

Research Goal: Develop an inert environment plasma arc welding (PAW) system and utilize it for fusion joining of ultra-high temperature ceramics

Current Status: Zirconium diboride (ZrB_2) has been successfully fusion bonded through PAW in an inert environment.

Research Challenges and Solutions

Oxidation

Problem: In an oxidizing environments, such as air, ZrB_2 will oxidize. During welding, oxidation can induce porosity in the melt fusion zone.

Solution: A glove box was modified with appropriate feed-through to act as an environmental chamber for arc welding of ZrB_2 .

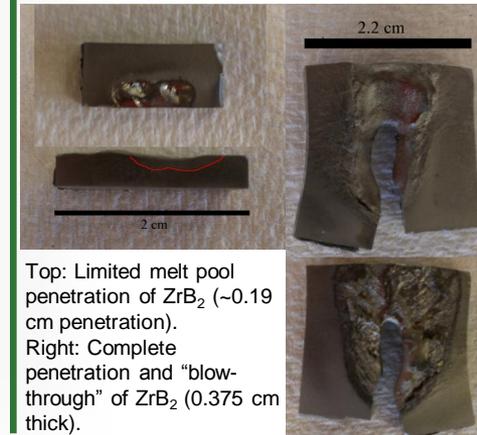
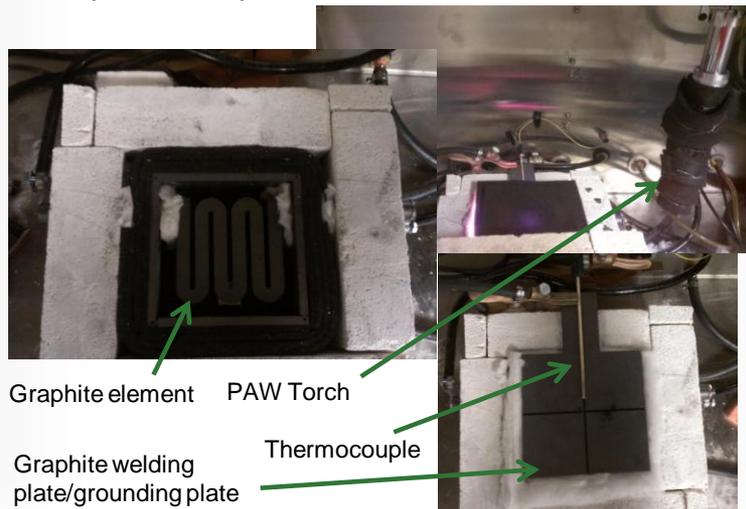
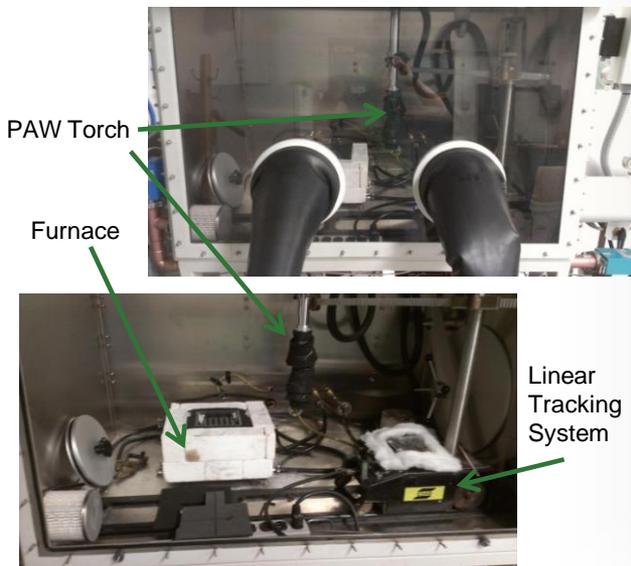
Thermal Shock

Problem: Metals have the ability to relieve thermal stress during welding due to their high fracture toughness and thermal conductivity values. ZrB_2 has a relatively low fracture toughness compared to metals, making it susceptible to thermal shock ($\Delta T_c=400^\circ C$). Welding can thermal shock ZrB_2 due to rapid heating when striking an arc.

Solution: A graphite element furnace is used to pre-heat ZrB_2 specimens for arc welding. Pre-heat temperatures up to $1525^\circ C$ have been utilized.

Current Progress

- Arc welding of ZrB_2 has been performed under inert environments at elevated temperatures.
 - Linear tracker controls weld speed
 - Weld sequencer controls current ramps and welding current
- Penetration of melt pool can be varied from slight penetration to full penetration.



Background: Joining Methods and Needs

Increasing Temperature

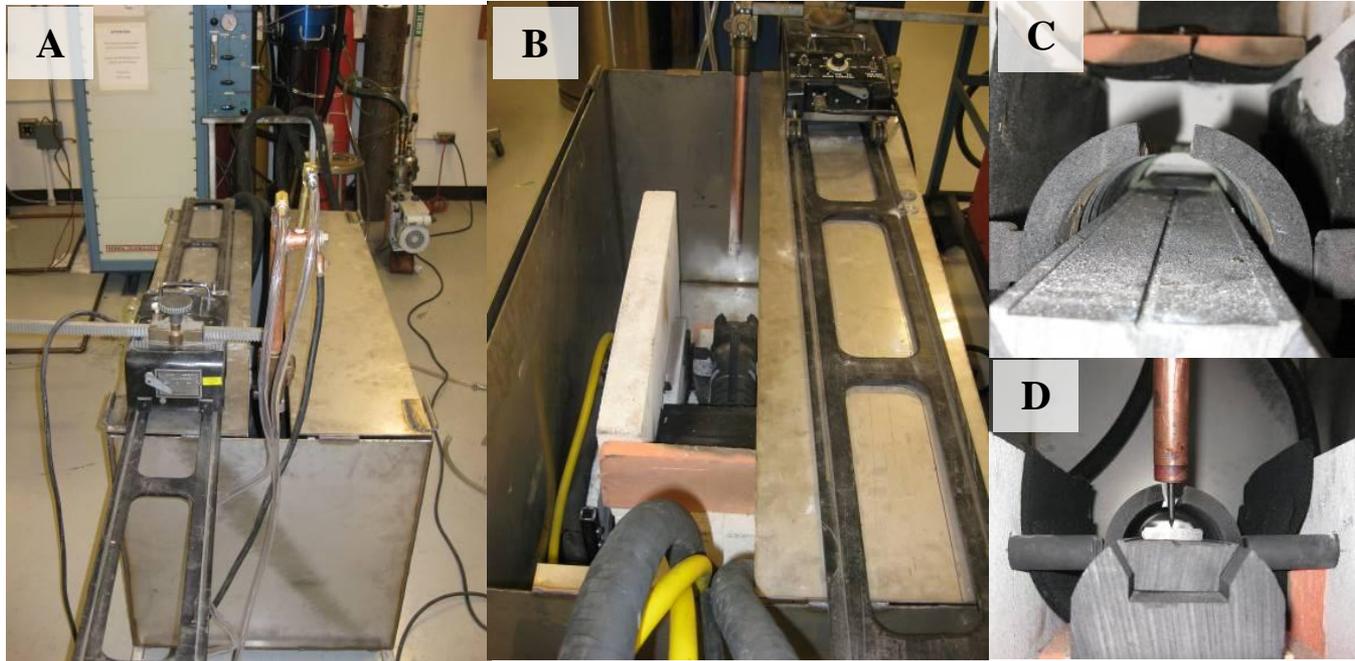


- Common joining methods
 - Polymers and cements
 - Glassy interlayers
Oxide and bulk metallic glass
 - Brazes
Non reactive alloys
Reactive
 - SHS joining
 - Mechanical fixtures
 - Diffusion Bonding
 - Plasma deposition
 - Fusion welding
E-beam, GTAW, plasma arc



- Considerations for TPS joining methods
 - Oxidation of joint and structure
 - Elevated temperature properties
 - CTE of parts
 - Manufacturability

Phase I: Preheating and Welding Setup

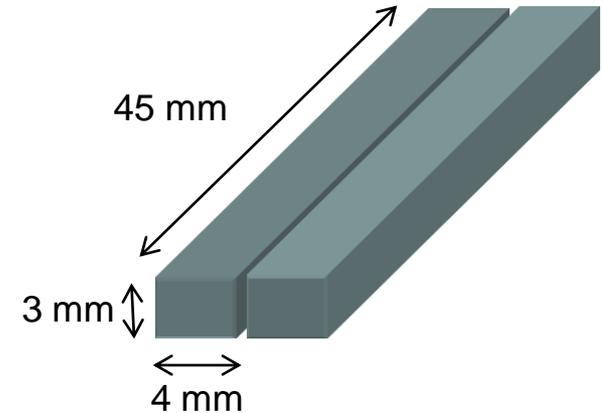


- **Welding chamber (A)**
 - 304 SS with graphite welding table
 - Split lid to contain Ar gas
- **Preheating chamber (C)**
 - 5 mm x up to 205 mm long specimens
 - Preheat up to 1700°C
- **Linear tracker (A & B)**
 - Moves water cooled copper stinger
 - 2 to 250 cm/min
- **Water cooled copper welding stinger (D)**
 - Holds 6.35 mm tungsten electrode

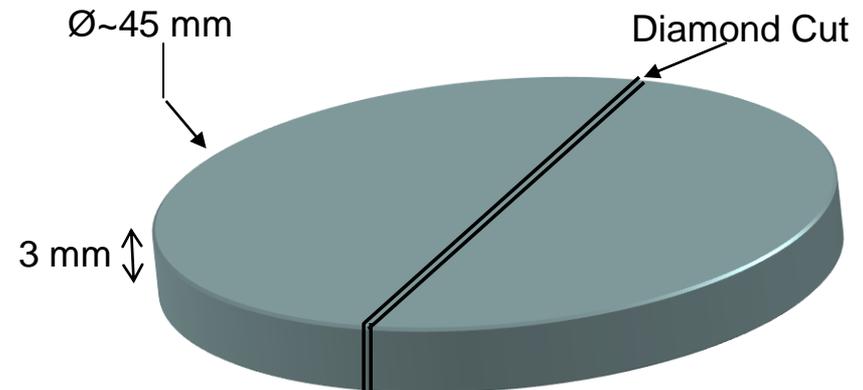
Phase I: Weld Specimens

- **Compositions**
 - $\text{ZrB}_2 + 20 \text{ vol\% SiC}$ (Z20S)
 - $\text{ZrB}_2\text{-SiC-B}_4\text{C}$ (ZSB)
- **Bars were cut and ground to size and then butt welded**
- **Disks were sectioned**
 - Diamond saw
 - Cleaned and chamfered
- **Welding methods**
 - Gas tungsten arc welding (GTAW)
 - Plasma arc welding (PAW)

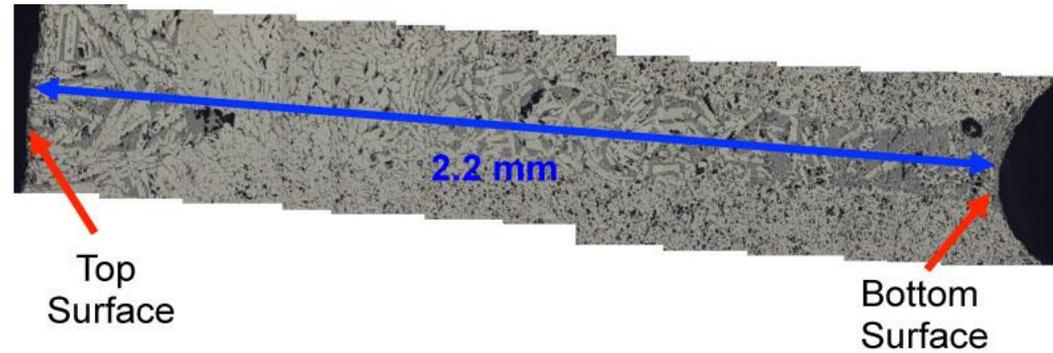
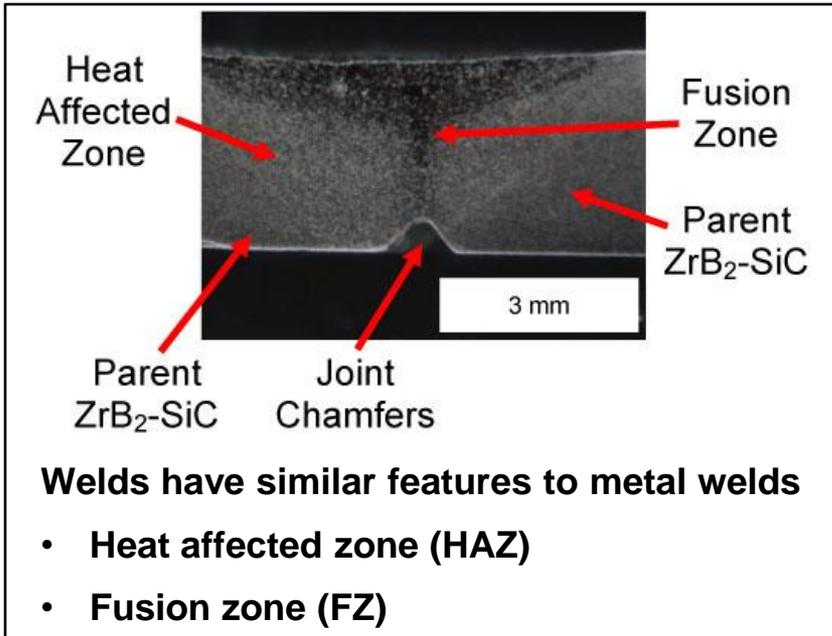
Butt Welded Bars



Butt Welded Discs

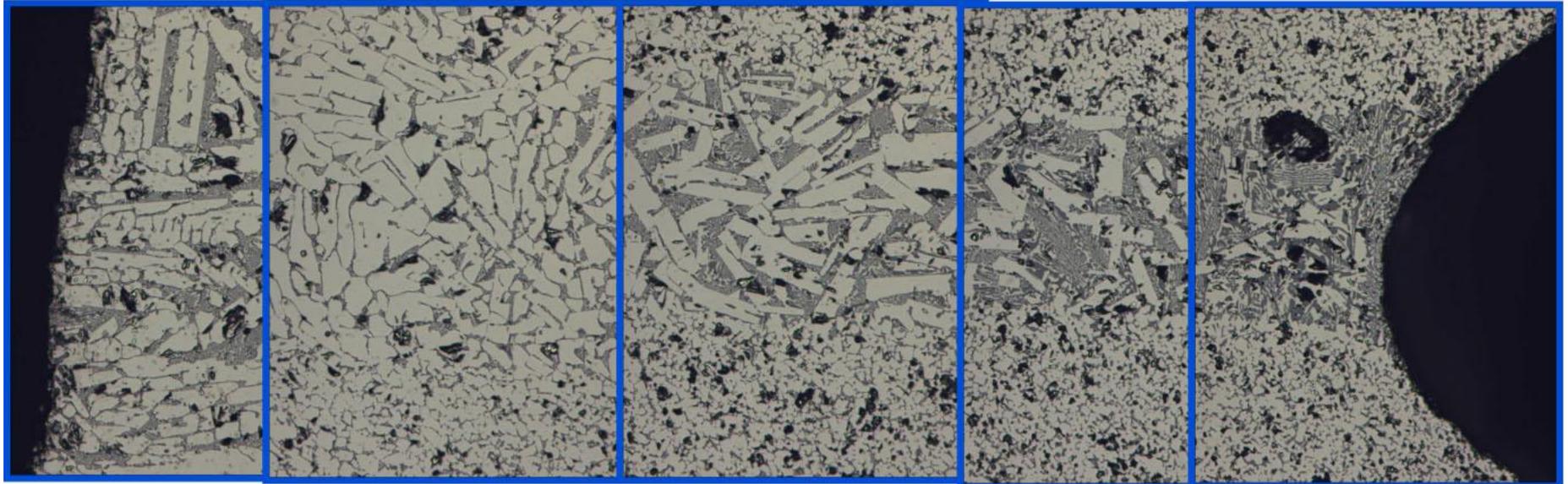


Phase I: GTAW Weld Pool Microstructure



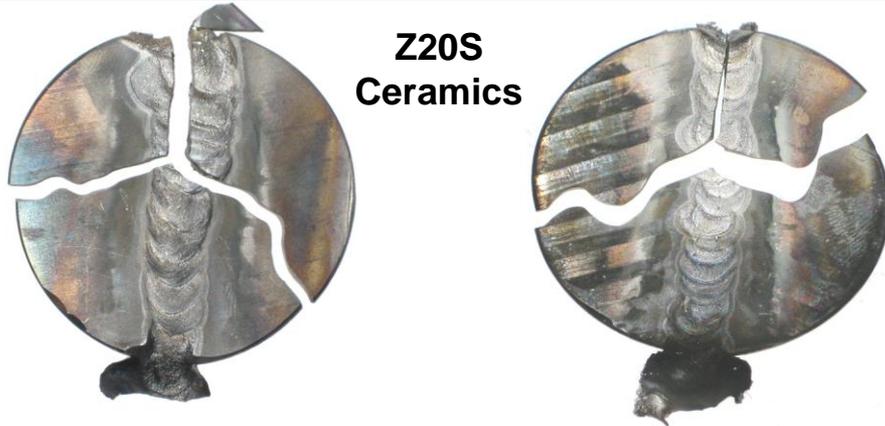
Stitched SEM images of the weld show ZrB_2 (bright phase) and SiC (gray phase):

- SiC appears to be partially depleted in HAZ
- Some porosity is visible



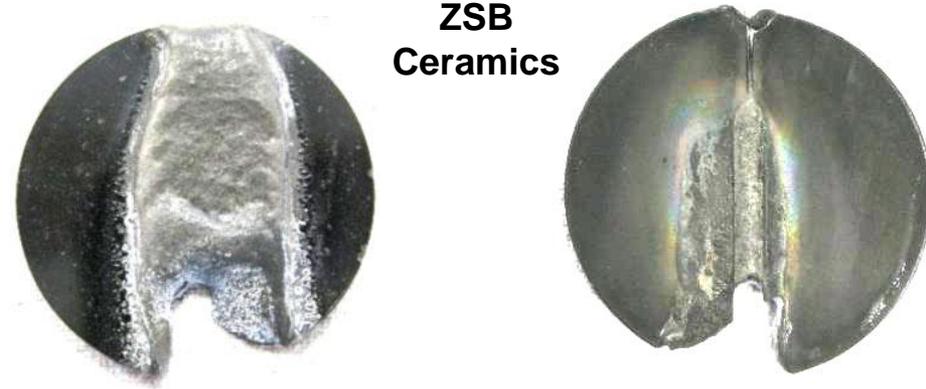
Phase I: Summary of Results

**Z20S
Ceramics**

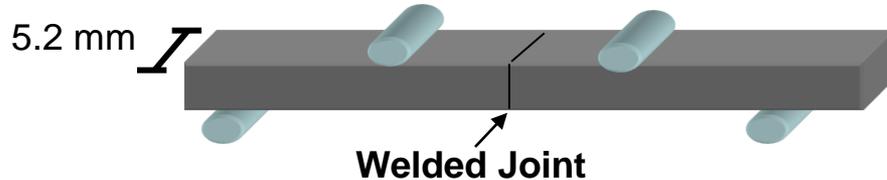


- Thermal stresses can lead to cracking
 - Controlled by sample pre- & post-heating

**ZSB
Ceramics**

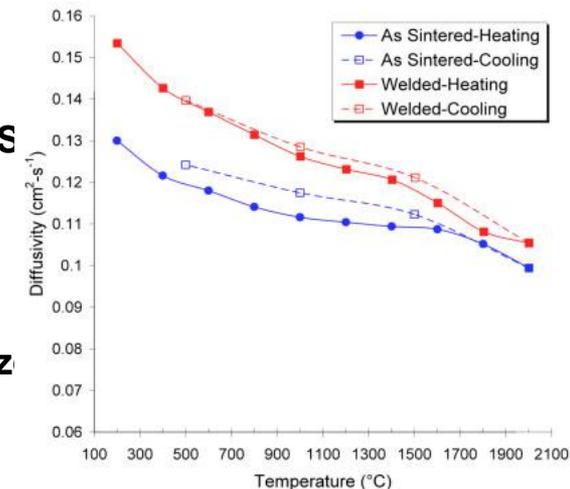


- Full penetration welds, without thermal cracking, were achieved with the correct heating/welding conditions



- Mechanical properties of the welded joint are controlled by residual stresses and porosity
 - <200 MPa for joined specimens compared to ~800 MPa for Z20S
 - Void formation at the HAZ-parent material interface

- Thermal diffusivity increases after welding
 - 0.13 cm²/s at 200° C for Z20S
 - 0.155 cm²/s at after welding
- Welding alters ceramic
 - Larger grain size
 - SiC polytype changes 6H to 3C



Phase II: Objectives

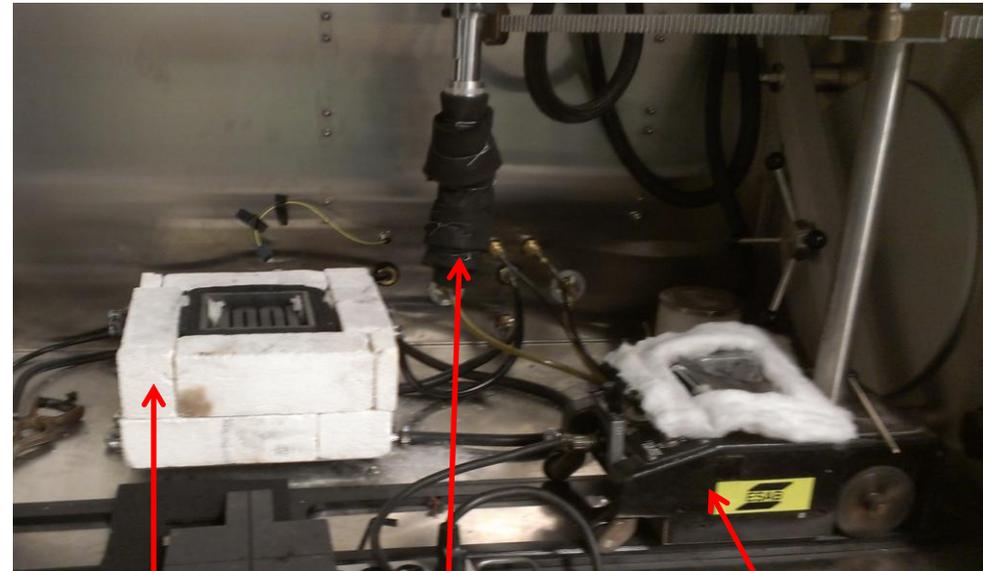
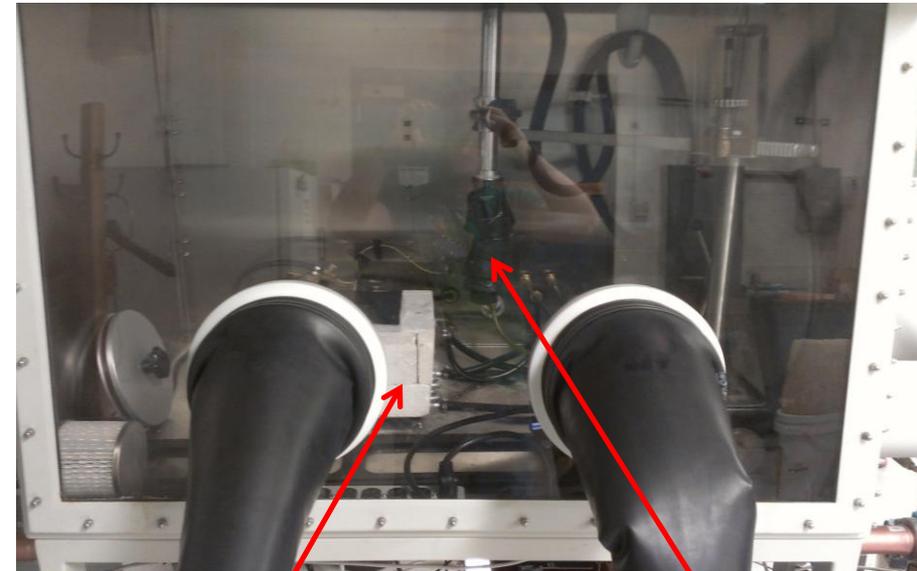
- **Develop second generation ceramic welding unit for technology transfer from Missouri S&T to MO-SCI**
- **Develop novel strategies to remove fusion weld discontinuities in UHTCs**
- **Complete macrostructure and microstructure characterization of welded UHTCs**
- **Measure thermo-mechanical and thermo-physical behavior and relate microstructural and macrostructure features to specific material response including fracture resistance, thermal management, and oxidation behavior of UHTCs**
- **Test a series of welded structures in an inductively coupled plasma heater facility (Prof. Fletcher – Univ. of Vermont) to better understand if UHTC weldments can withstand the aggressive thermal and oxidative environment associated with hypersonic flight**

Phase II: Inert Environment Chamber

- An M-Braun Glovebox was modified to hold the necessary tools for performing plasma-arc welding of UHTC's
- Chamber holds pre-heating furnace, PAW torch, and linear tracking system

Glovebox with front panel

Glovebox without front panel



Pre-heat Furnace

PAW torch

Pre-heat Furnace

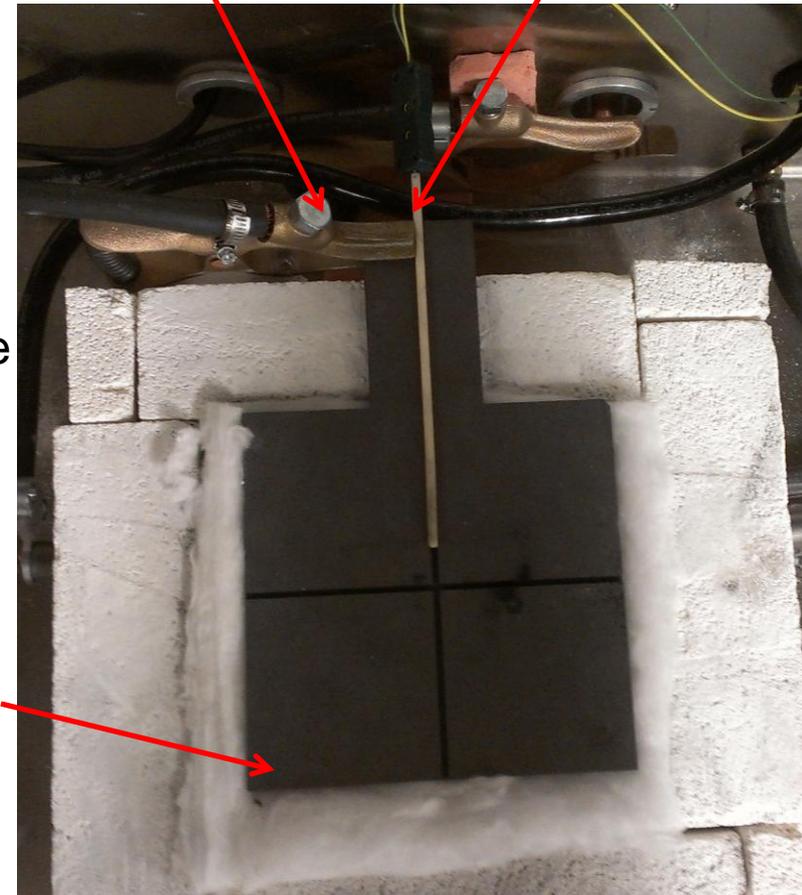
PAW torch

Linear Tracking System

Phase II: Pre-heating Furnace

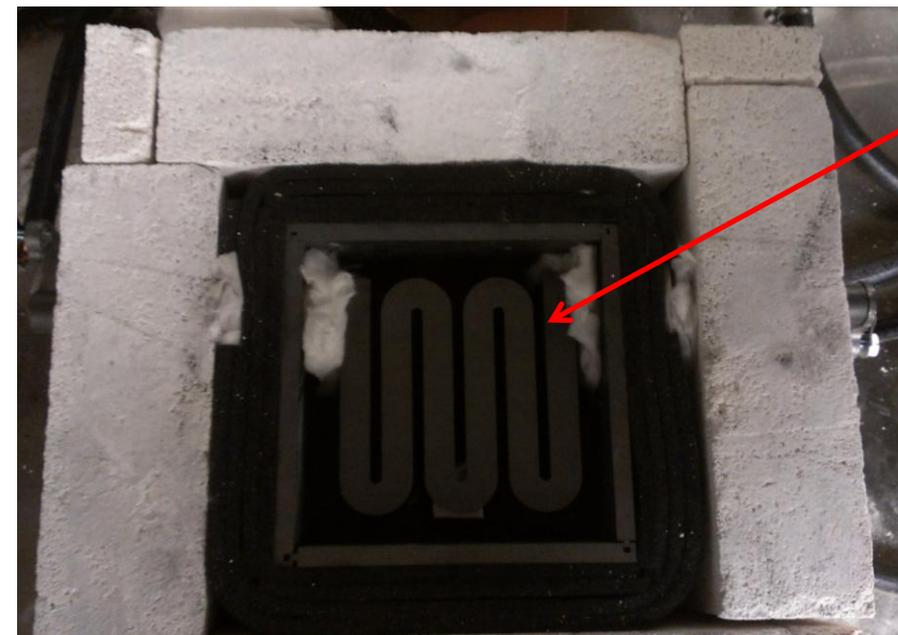
- Graphite element furnace
 - Pre-heat temperatures of 1550°C
 - 4x4" hot zone
 - Controlled ramp up and cool down
 - Graphite lid for welding ground

Type B thermocouple
Welding ground



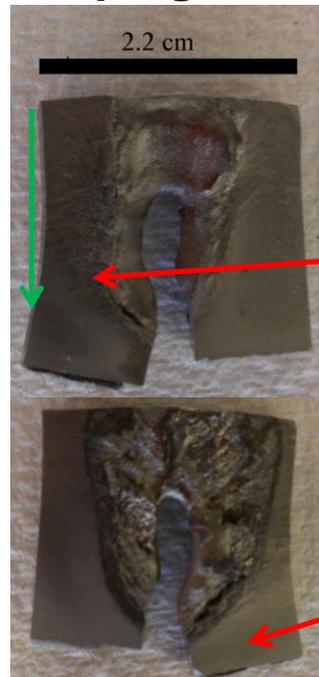
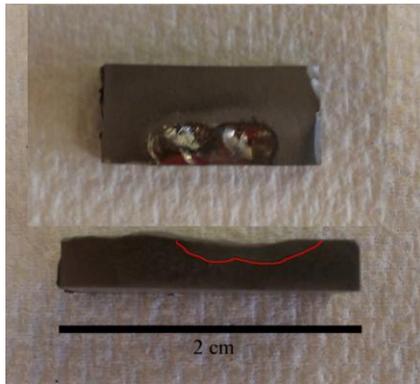
Graphite element

Graphite welding plate



Phase II: Welding of ZrB_2

- ZrB_2 base was pre-heated to $1450^\circ C$ or higher
- Varying penetration achieved
- High thermal stresses due to high melting point of ZrB_2
 - $>3000^\circ C$
 - Cracking and warping have been observed

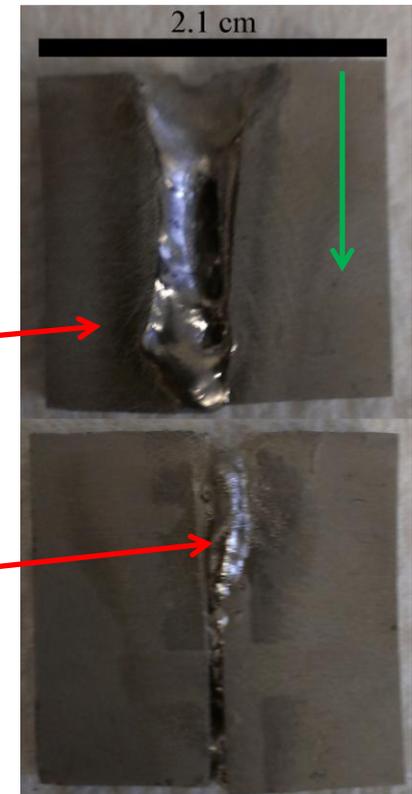


Weld direction indicated by green arrows

Cracking along weld due to high thermal stresses

Full penetration of joint

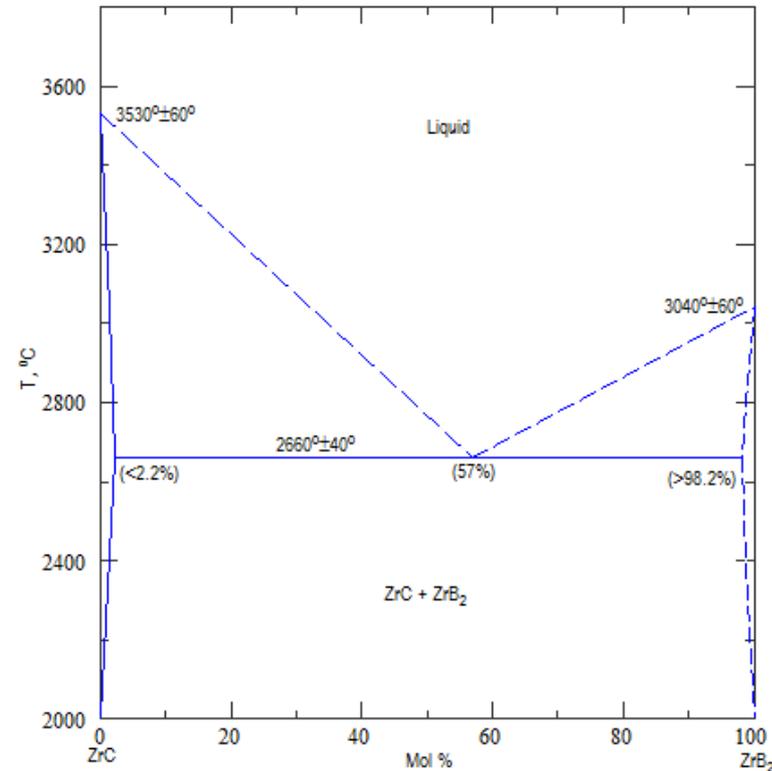
Thermal warping of ZrB_2



Top: Limited melt pool penetration of ZrB_2 (~0.19 cm penetration).
 Right: Complete penetration and "blow-through" of ZrB_2 (0.375 cm thick).

Phase II: Diboride-Carbide Eutectics

- Diboride-Carbide eutectics have lower melting temperatures than the pure diboride
- Decreases thermal gradient
- Possible compositions
 - $\text{ZrB}_2\text{-ZrC}$, $T_M=2660^\circ\text{C}$
 - $\text{TiB}_2\text{-TiC}$, $T_M=2520^\circ\text{C}$
 - $\text{ZrB}_2\text{-SiC}$, $T_M=2270^\circ\text{C}$
 - $\text{TiB}_2\text{-SiC}$, $T_M=2190^\circ\text{C}$



Phase II: Reactive Filler Production

- Candidate fillers:

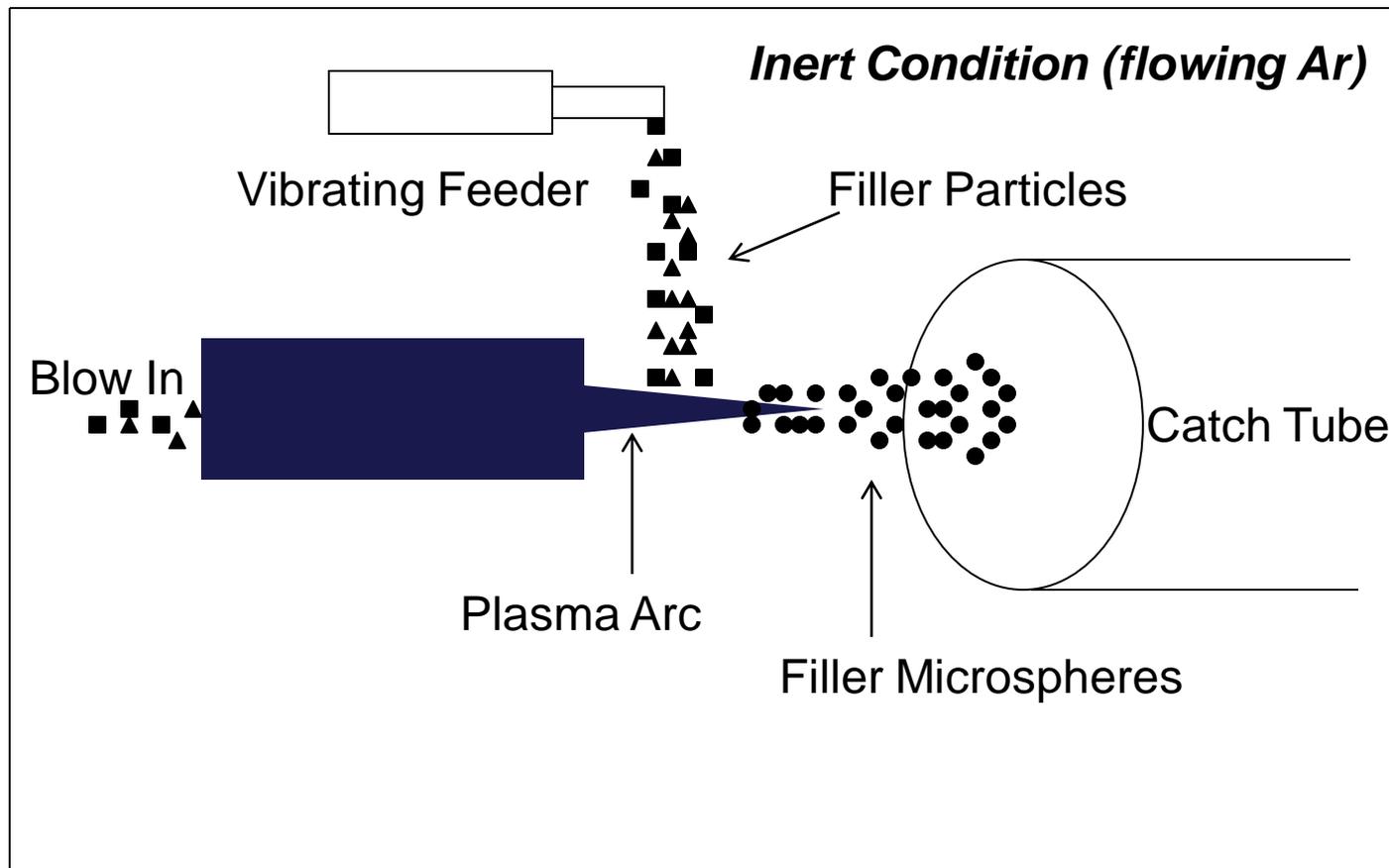
Reactive: High purity SiB_6 , ZrC , Zr \longrightarrow ZrB_2 -20 v/o SiC

Excess reactive: Mg- or Zr-alloys \longrightarrow MgO or ZrO_2 precipitates
(i.e., deoxidize)

- Form: microspheres \longrightarrow easy to apply through the PAW system
- Size: <75 microns
- Plasma arc “Spheridization” using “old welding system”

Phase II: Reactive Filler Production-cont.

- Ultra high temperature ($>3000^{\circ}\text{C}$) spheridization system



Summary

- **Phase I (Completed)**
 - **ZrB₂-based ceramics up to 3 mm thick were joined by fusion welding**
 - **Joint microstructures varied through the part**
 - **Joint strengths (<200 Mpa) were lower than the parent materials (800 MPa), likely due to void formation**
 - **Thermal conductivity of joints was higher than parent material**
- **Phase II (In progress)**
 - **Second generation ceramic welding unit designed and built at MS&T**
 - **Welding technology being transferred from MS&T to MO-SCI**
 - **MS&T welding ZrB₂-based UHTCs with efforts focused on eliminating porosity in the welds and obtaining full weld penetration**
 - **MO-SCI developing technologies for filler materials to add to the PAW systems at both MS&T and MO-SCI**