

# **Welding of $\text{ZrB}_2$ -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 ( $\text{ZrB}_2$ ) has been successfully fusion bonded through PAW in an inert environment.

## Research Challenges and Solutions

### Oxidation

**Problem:** In an oxidizing environments, such as air,  $\text{ZrB}_2$  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  $\text{ZrB}_2$ .

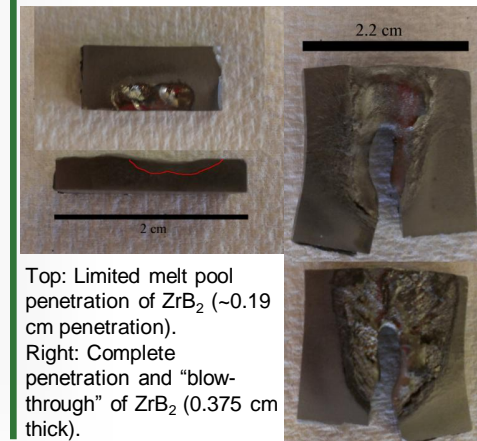
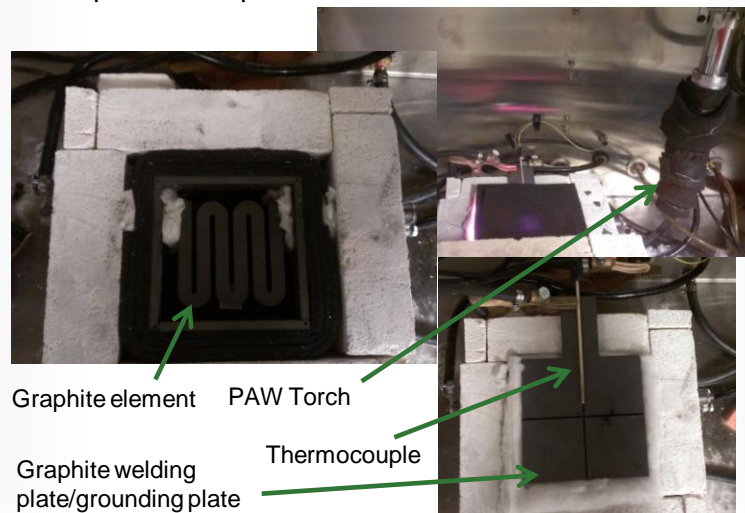
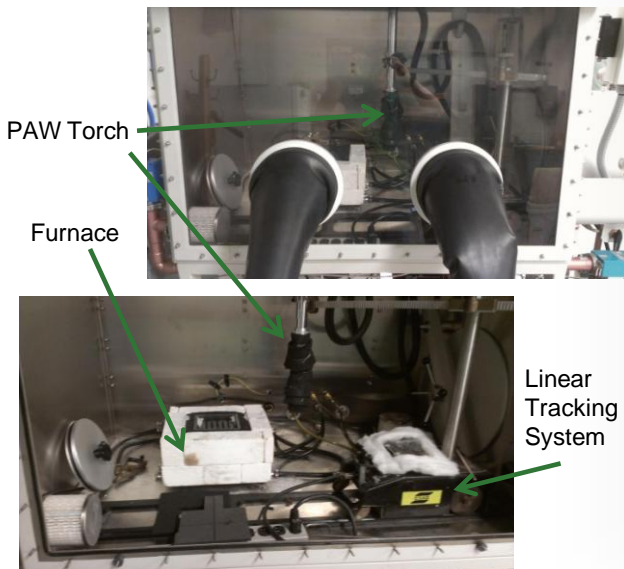
### Thermal Shock

**Problem:** Metals have the ability to relieve thermal stress during welding due to their high fracture toughness and thermal conductivity values.  $\text{ZrB}_2$  has a relatively low fracture toughness compared to metals, making it susceptible to thermal shock ( $\Delta T_c = 400^\circ \text{C}$ ). Welding can thermal shock  $\text{ZrB}_2$  due to rapid heating when striking an arc.

**Solution:** A graphite element furnace is used to pre-heat  $\text{ZrB}_2$  specimens for arc welding. Pre-heat temperatures up to  $1525^\circ \text{C}$  have been utilized.

### Current Progress

- Arc welding of  $\text{ZrB}_2$  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

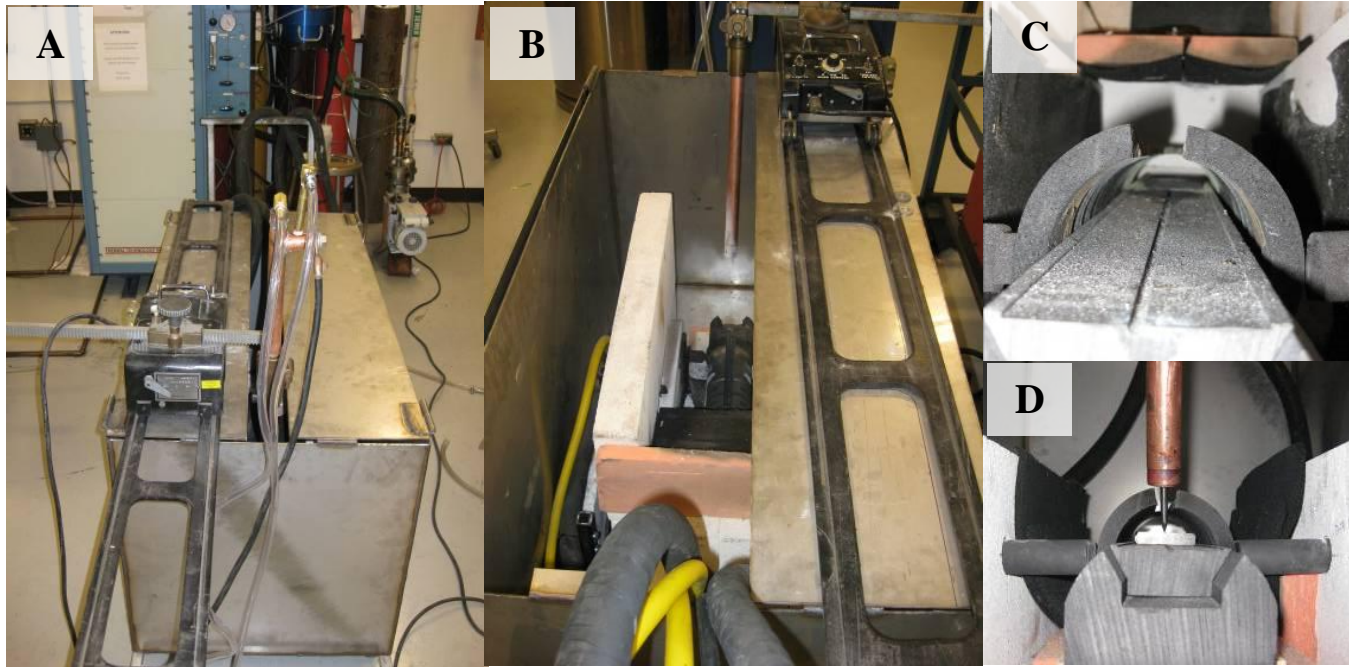


- Common joining methods
  - Polymers and cements
  - Glassy interlayers  
Oxide and bulk metallic glass
  - Brazes  
Non reactive alloys  
Reactive
  - SHS joining
  - Mechanical fixtures
  - Diffusion Bonding
  - Plasma deposition
  - Fusion welding  
E-beam, GTAW, plasma arc



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

# Phase I: Preheating and Welding Setup



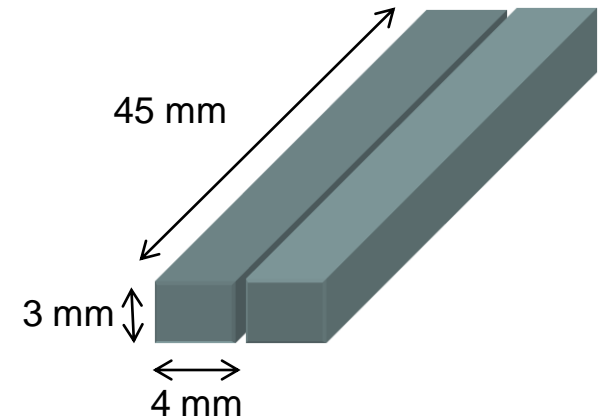
- **Welding chamber (A)**
  - 304 SS with graphite welding table
  - Split lid to contain Ar gas
- **Preheating chamber (C)**
  - 5 mm x up to 205 mm long specimens
  - Preheat up to 1700°C
- **Linear tracker (A & B)**
  - Moves water cooled copper stinger
  - 2 to 250 cm/min
- **Water cooled copper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



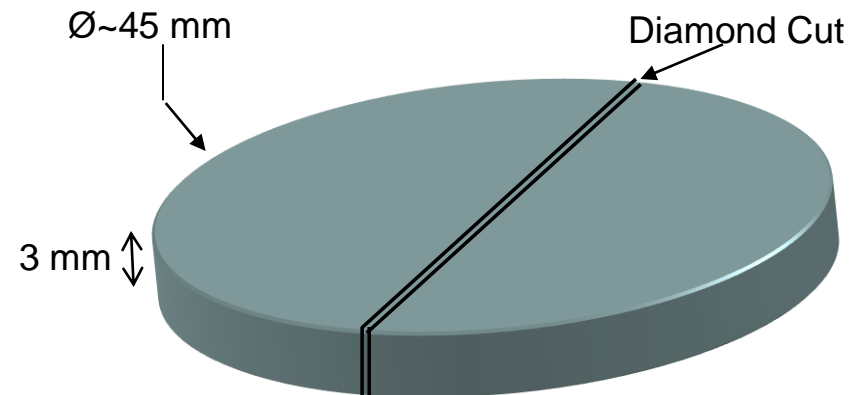
# Phase I: Weld Specimens

- **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)

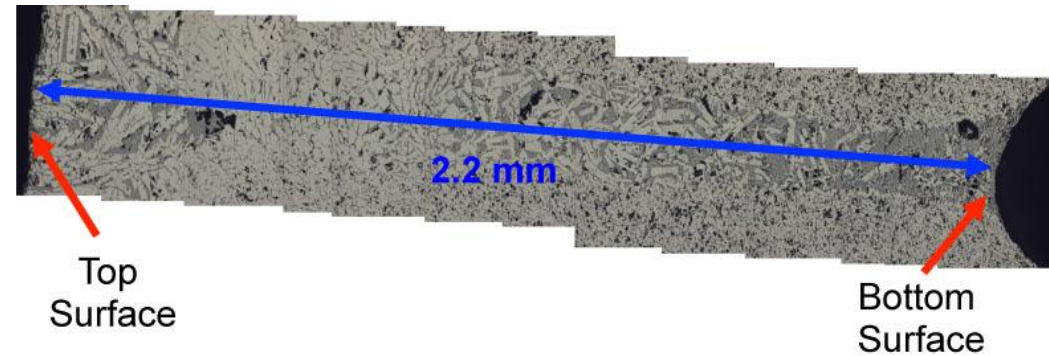
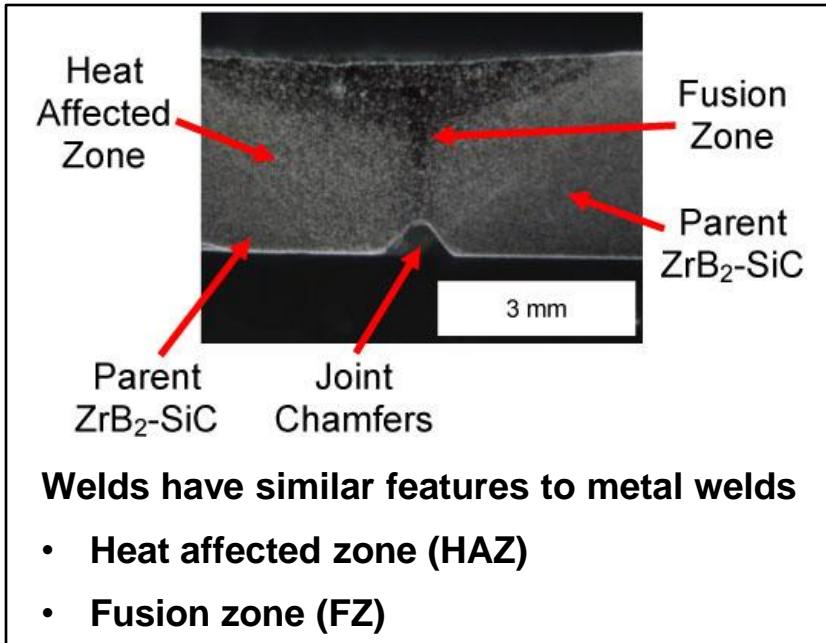
## Butt Welded Bars



## Butt Welded Discs

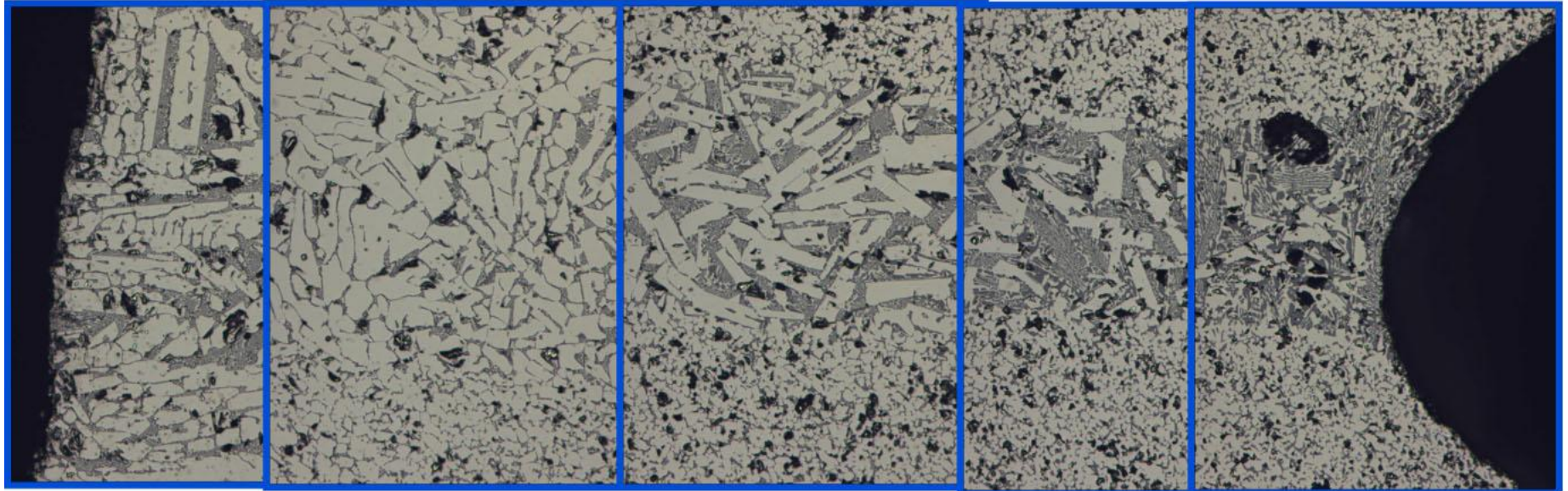


# Phase I: GTAW Weld Pool Microstructure



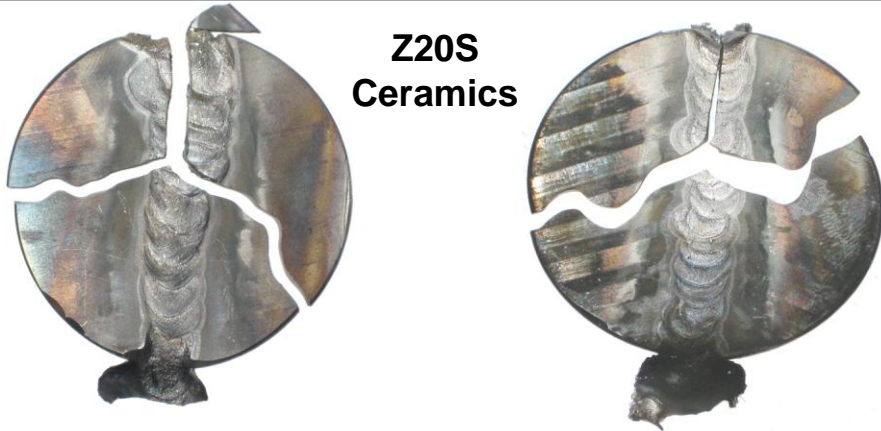
Stitched SEM images of the weld show  $\text{ZrB}_2$  (bright phase) and SiC (gray phase):

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



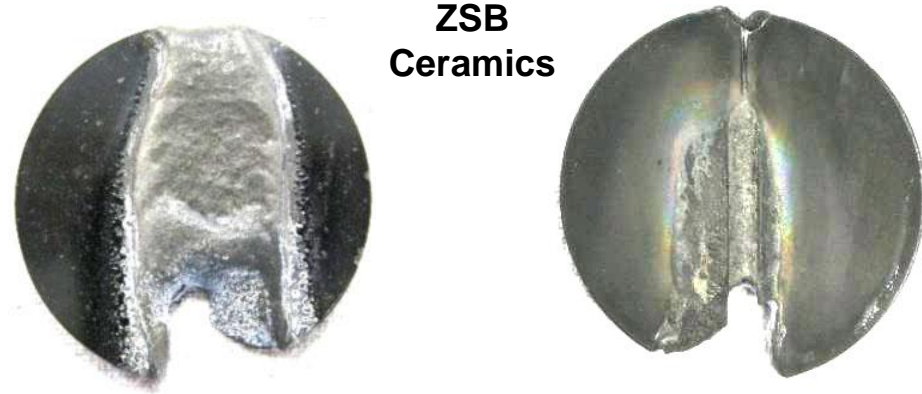
# Phase I: Summary of Results

**Z20S  
Ceramics**

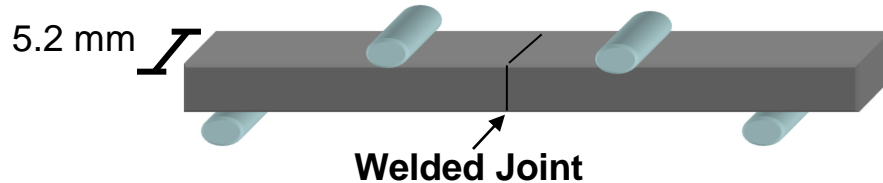


- Thermal stresses can lead to cracking
  - Controlled by sample pre- & post-heating

**ZSB  
Ceramics**

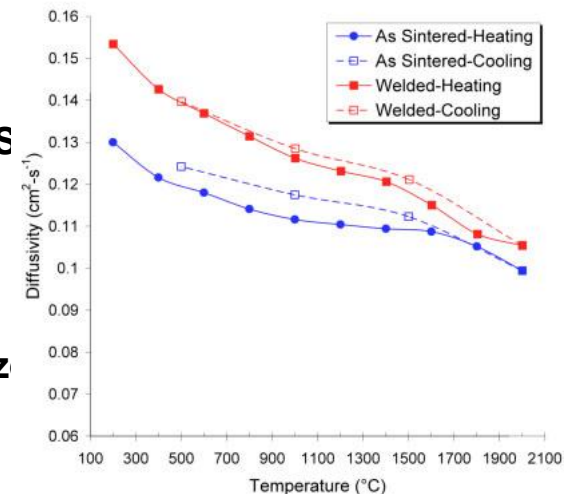


- Full penetration welds, without thermal cracking, were achieved with the correct heating/welding conditions



- 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 cm<sup>2</sup>/s at 200° C for Z20S
  - 0.155 cm<sup>2</sup>/s at after welding
- Welding alters ceramic
  - Larger grain size
  - SiC polytype changes 6H to 3C





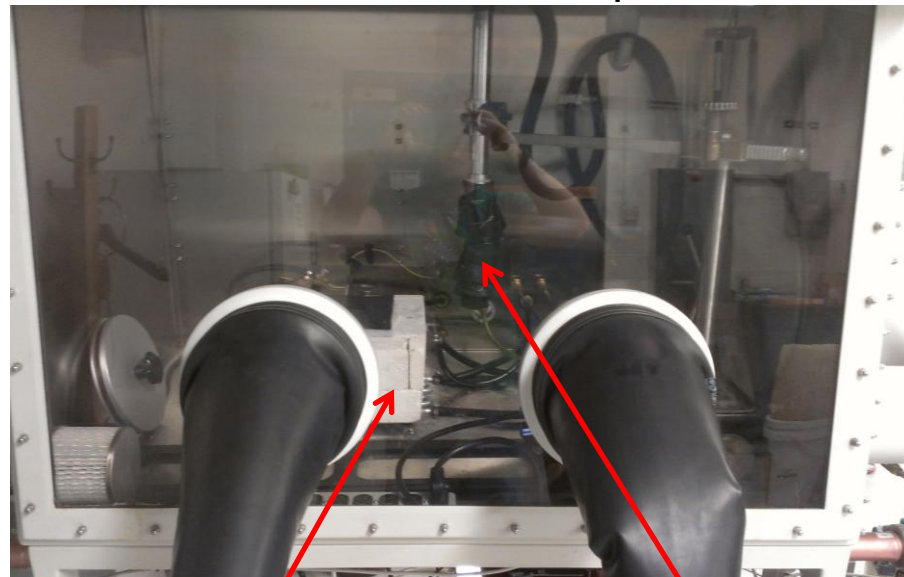
## Phase II: Objectives

- **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

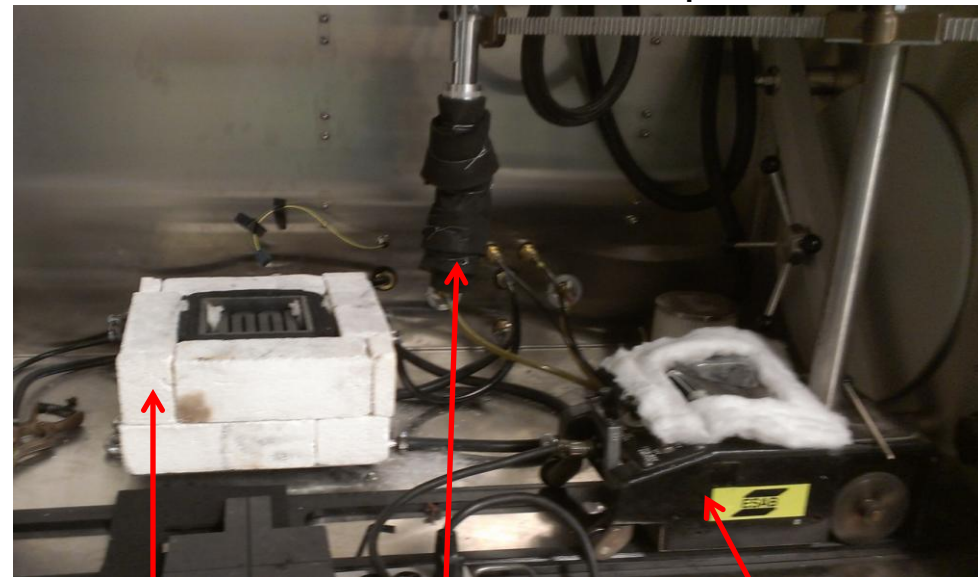
Glovebox with front panel



Pre-heat Furnace

PAW torch

Glovebox without front panel



Pre-heat Furnace

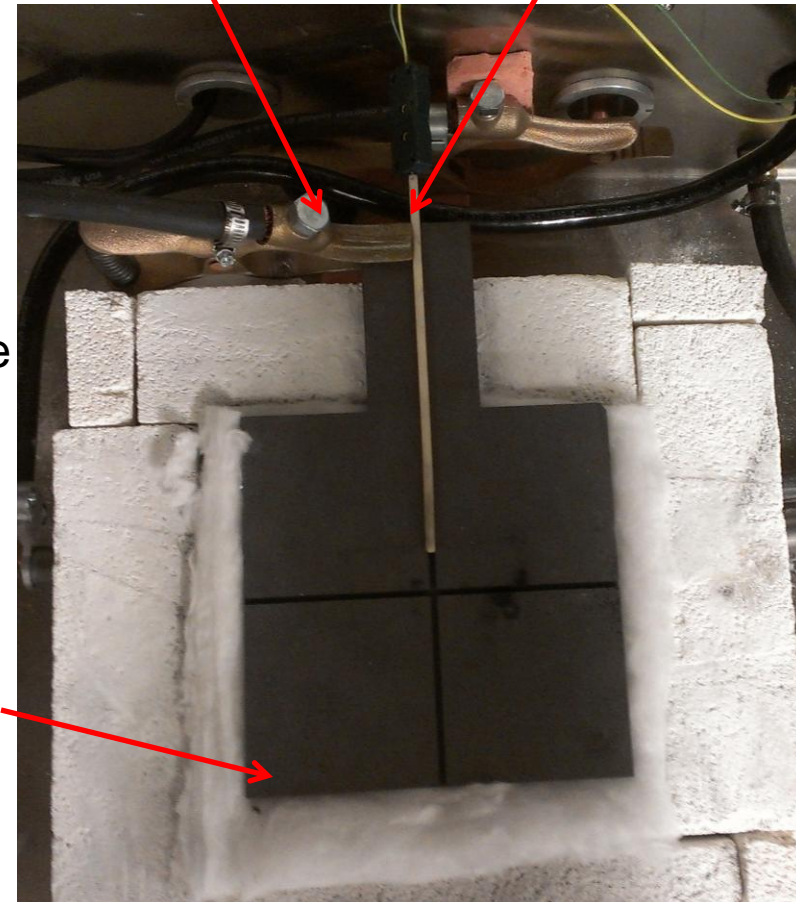
PAW torch

Linear Tracking System

# Phase II: Pre-heating Furnace

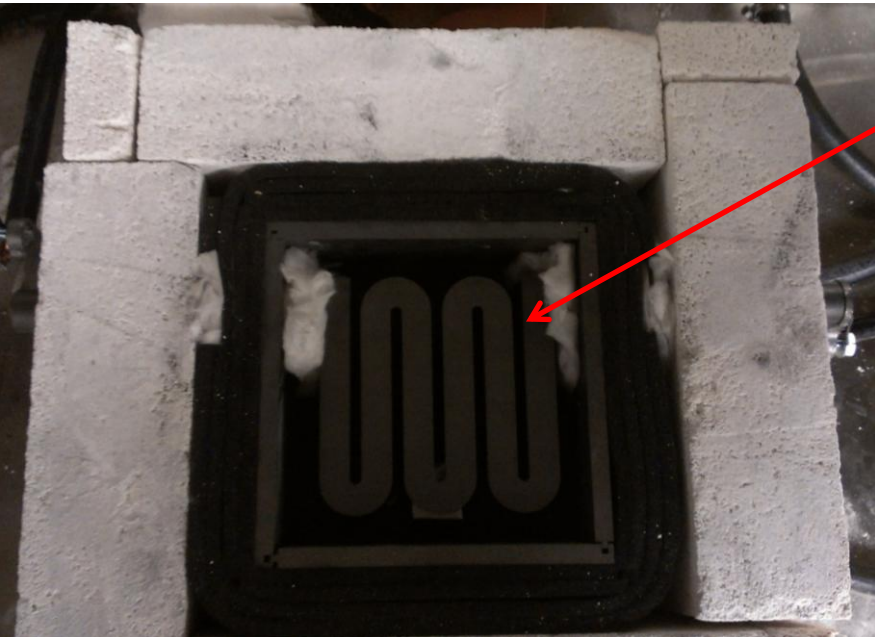
- Graphite element furnace
  - Pre-heat temperatures of 1550°C
  - 4x4" hot zone
  - Controlled ramp up and cool down
  - Graphite lid for welding ground

Type B thermocouple  
Welding ground



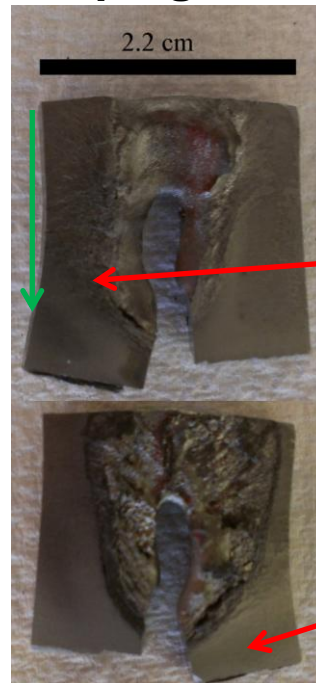
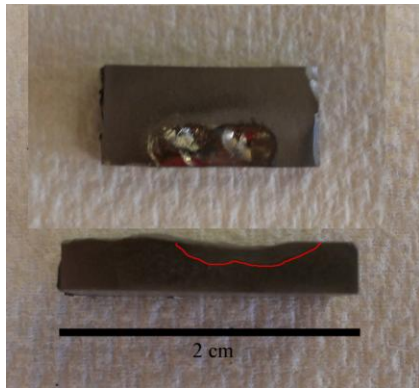
Graphite element

Graphite welding plate



# Phase II: Welding of $\text{ZrB}_2$

- $\text{ZrB}_2$  base was pre-heated to  $1450^\circ\text{C}$  or higher
- Varying penetration achieved
- High thermal stresses due to high melting point of  $\text{ZrB}_2$ 
  - $>3000^\circ\text{C}$
  - Cracking and warping have been observed

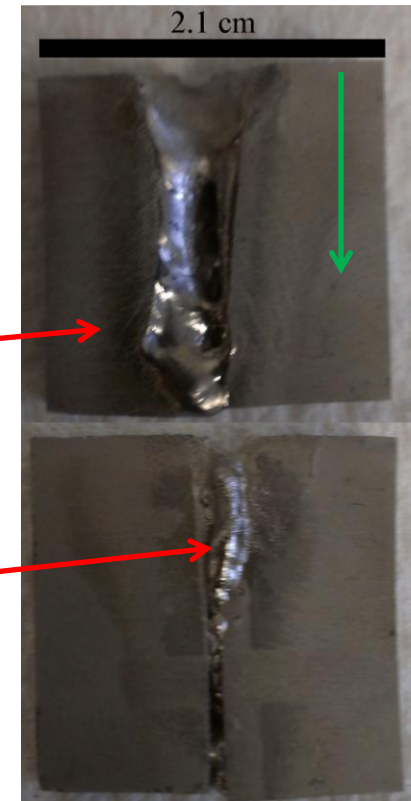


Weld direction indicated by green arrows

Cracking along weld due to high thermal stresses

Full penetration of joint

Thermal warping of  $\text{ZrB}_2$

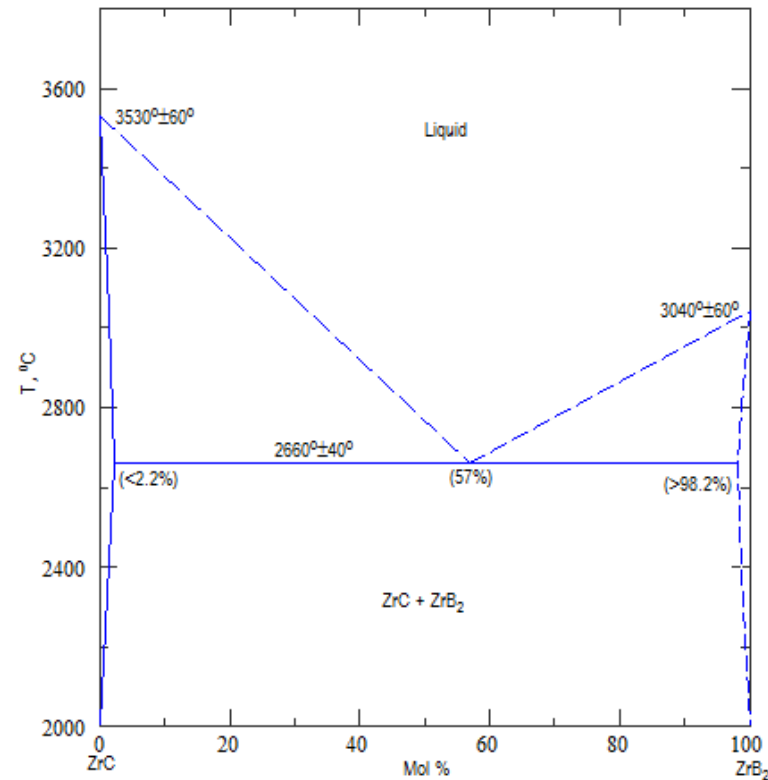


Top: Limited melt pool penetration of  $\text{ZrB}_2$  (~0.19 cm penetration).  
Right: Complete penetration and "blow-through" of  $\text{ZrB}_2$  (0.375 cm thick).



# 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:**

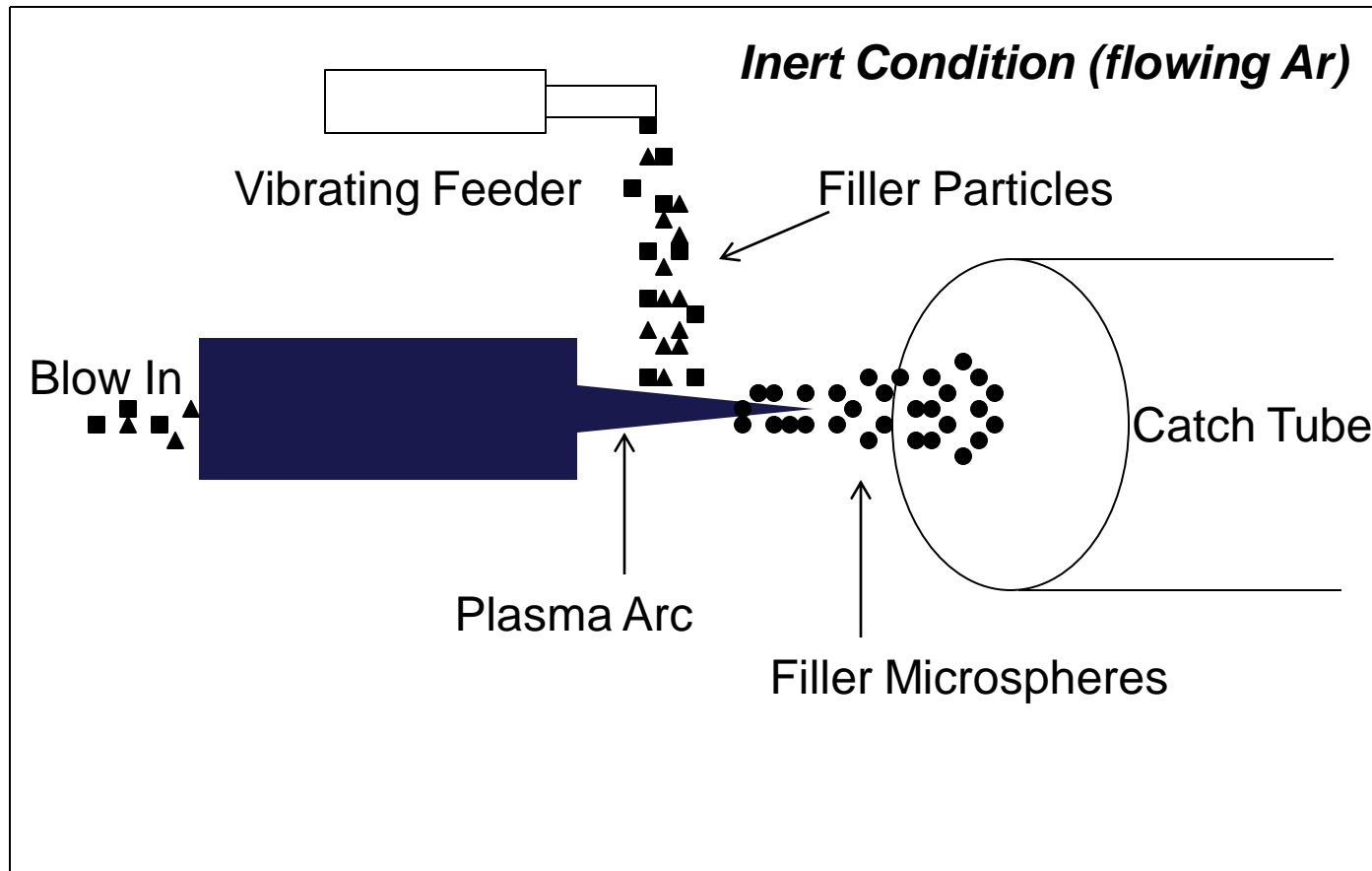
**Reactive: High purity  $\text{SiB}_6$ ,  $\text{ZrC}$ ,  $\text{Zr}$        $\longrightarrow$        $\text{ZrB}_2$ -20 v/o  $\text{SiC}$**

**Excess reactive: Mg- or Zr-alloys       $\longrightarrow$        $\text{MgO}$  or  $\text{ZrO}_2$  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)**
  - **ZrB<sub>2</sub>-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 ZrB<sub>2</sub>-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**