

# FW Joining Technology

## Be/CuCrZr Joining Development

S.H. Goods<sup>1</sup>, J.D. Puskar<sup>2</sup>

*Sandia National Laboratories,  
1Livermore, CA, USA, 94550 and  
2Albuquerque, NM, USA, 87185*

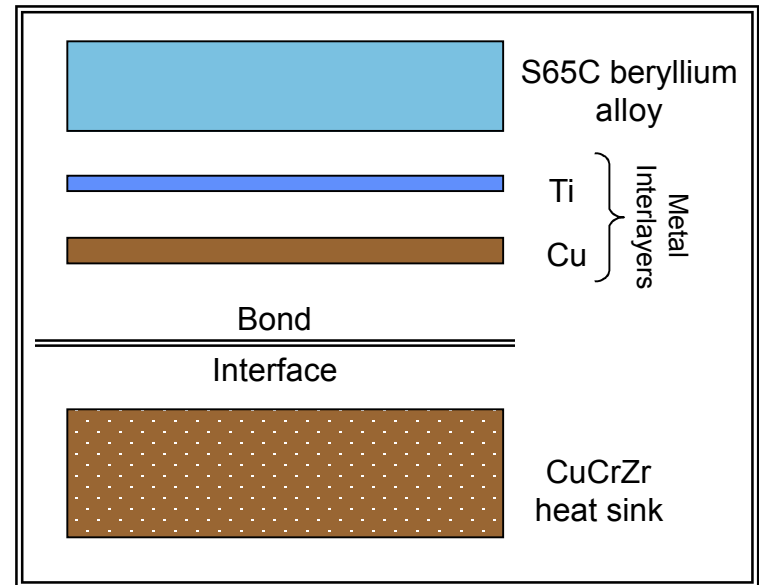
# Be/CuCrZr Joining Development

---

- US efforts directed at minimizing Be/CuCrZr HIP bonding temperature
  - To meet ITER defined CuCrZr strength requirements
    - Yield/UTS = 175/280 MPa @ RT
    - Yield/UTS = 150/220 MPa @ 250C
  - Maintain high strength and good consistency in bond quality

# Material preparation

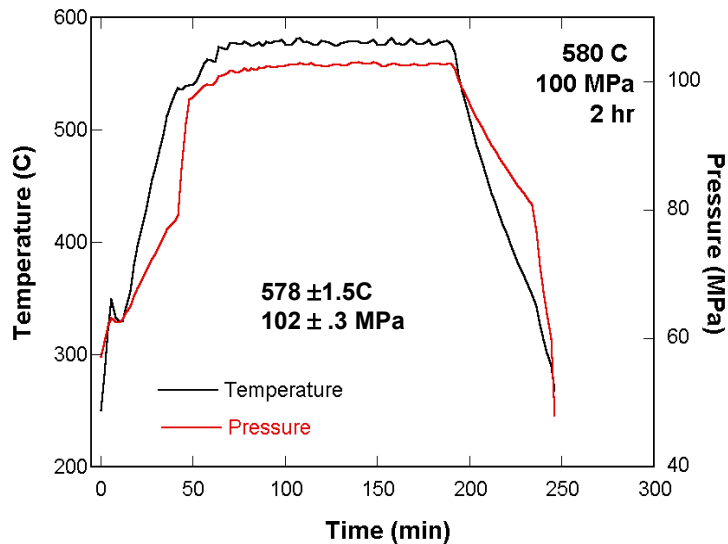
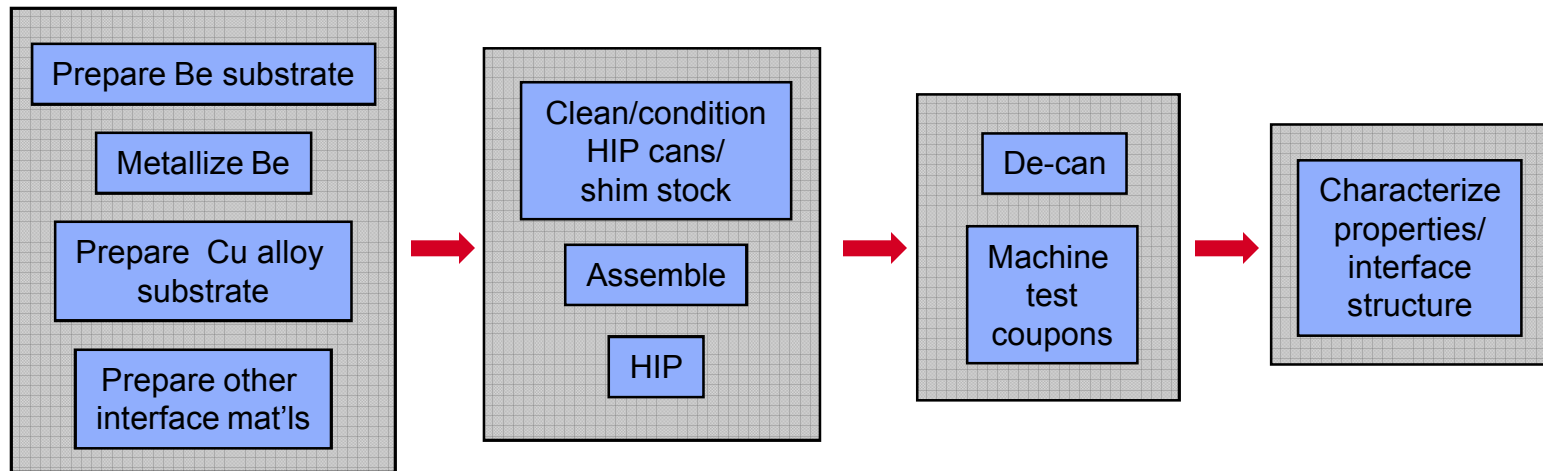
- CuCrZr and Be substrates (5 cm dia. disks)
  - Mechanically prepared surfaces
  - Chemically cleaned
- Interlayer metallizations
  - PVD processes
    - Sputter deposition or E-beam evaporation.
      - Titanium is applied to act as a barrier between Be and Cu suppressing the formation of brittle Be/Cu intermetallics.
      - Thick (25  $\mu\text{m}$ ) Cu metallization is applied as a mechanical compliance layer
  - In some instances free standing foils of copper are used in place of thick Cu metallization films.



## HIP processing conditions:

- Pressure: 100 MPa
- Time: 2 hr
- Temperature: 540C  $\rightarrow$  580 C

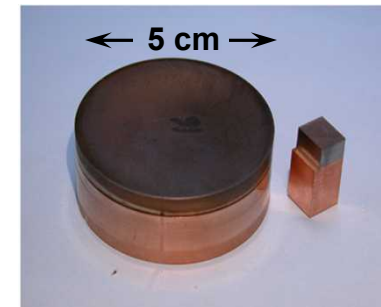
# HIP Process Flow



Typical temperature/pressure traces for HIP bonding cycle



HIP'ped container




HIP bonded Be and Cu alloy disk along with shear specimen

# Metallization/bonding schemes- sputter deposition

Be Surface Prep.	Metallization Process	Diffusion Barrier	Compliant Layer	CuCrZr Surface Preparation	HIP Temp (C)
(ga)(s)	Sputter Coat	2 $\mu$ m Ti	25 $\mu$ m Cu	Chem. Polish	580
(ga)(ns)	Sputter Coat	2 $\mu$ m Ti	25 $\mu$ m Cu	Chem. Polish	580
(ga)(s)	Sputter Coat	2 $\mu$ m Ti	3 $\mu$ m Cu-50 $\mu$ m Cu(f)	Chem. Polish	580
(ga)(s)	Sputter Coat	2 $\mu$ m Ti	25 $\mu$ m Cu	Chem. Polish	560
(ga)(s)	Sputter Coat	2 $\mu$ m Ti	3 $\mu$ m Cu-50 $\mu$ m Cu(f)	Chem. Polish	560
(pl)(s)	Sputter Coat	2 $\mu$ m Ti	25 $\mu$ m Cu	Chem. Polish	560
(ga)(s)	Sputter Coat	2 $\mu$ m Ti	25 $\mu$ m Cu	Chem. Polish	540
(pl)(s)	Sputter Coat	2 $\mu$ m Ti	25 $\mu$ m Cu	Chem. Polish	540

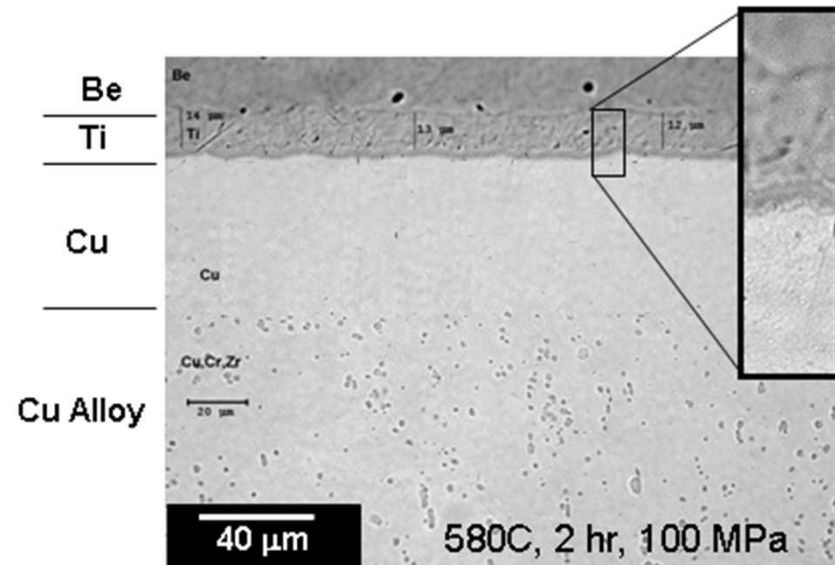
Decreasing  
HIP  
Temperature



(ga): grit abraded, chem. clean    (f): foil  
 (s): sputter clean    (pl): lapped  
 (ns): no sputter clean

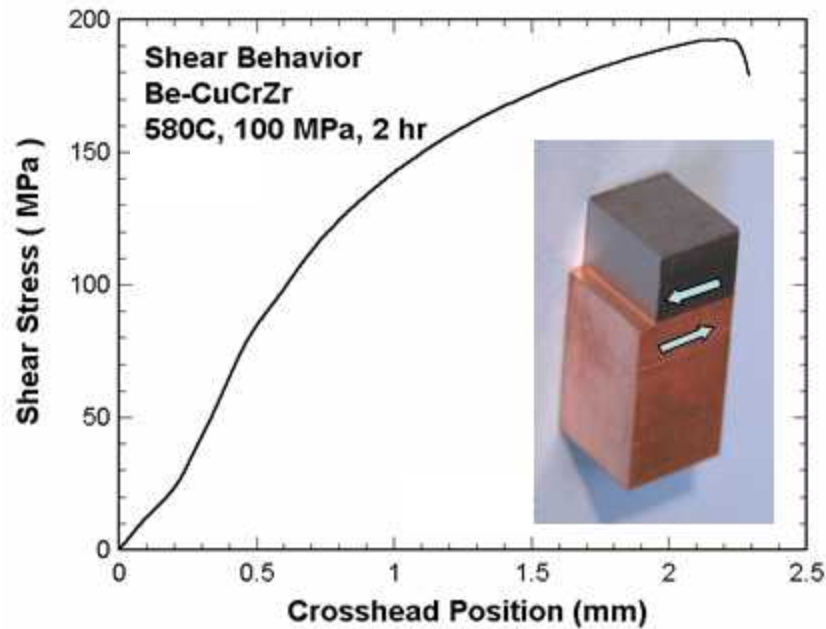
Focus is directed towards reducing HIP'ping temperature in order to increased margin on CuCrZr strength

# Optical cross-section of bond joint



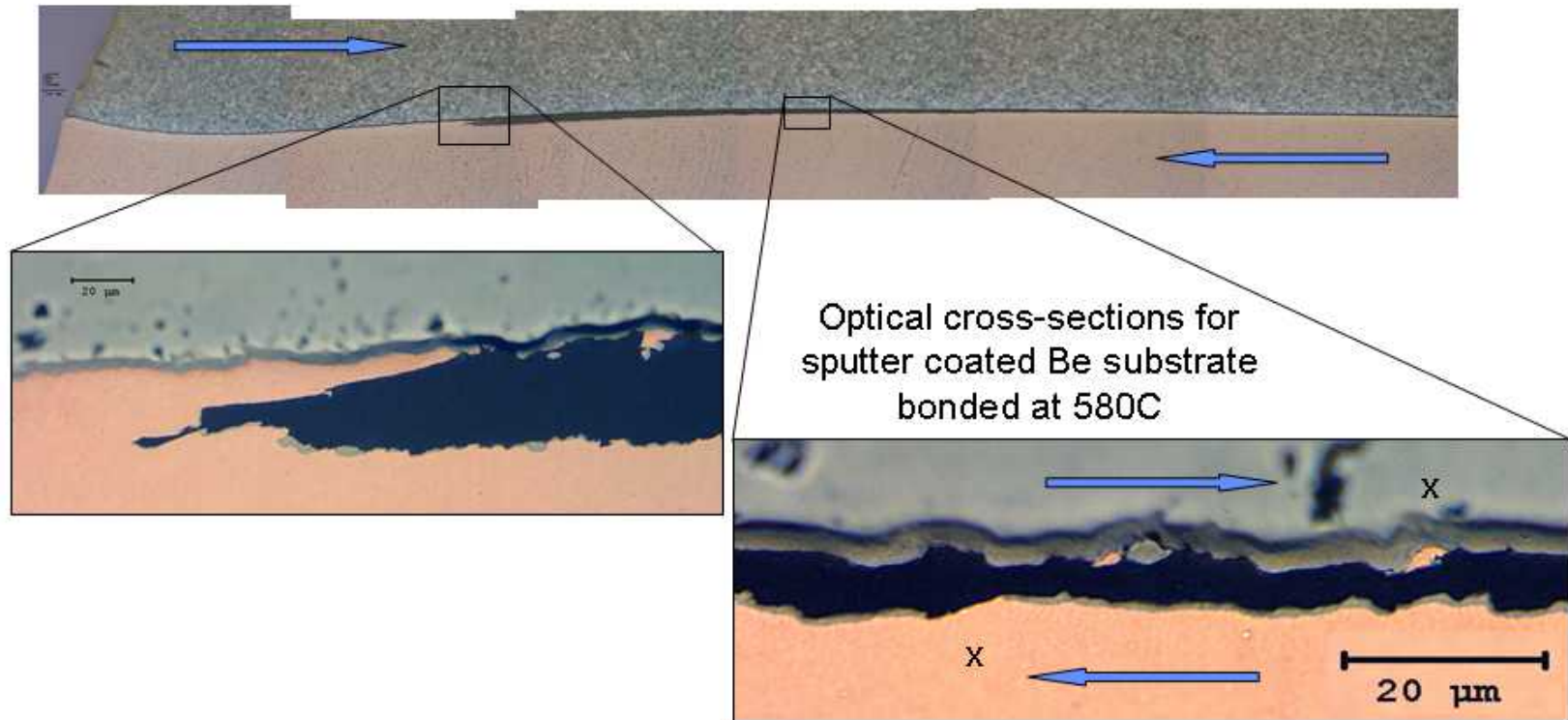
HIP processed bonds exhibit no interface defects

# Typical shear test



Shear strength is the principal measurement of bond quality

# Interrupted shear test

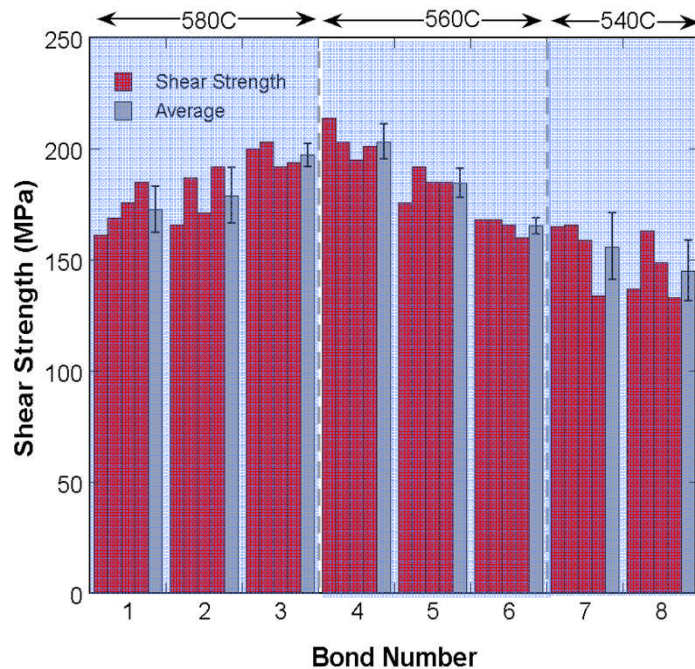


Thick Cu metallization is an effective compliance layer, allowing the joint to accommodated substantial deformation prior to failure

Fiducial marks ("x") indicate the relative shear offset within the internal crack

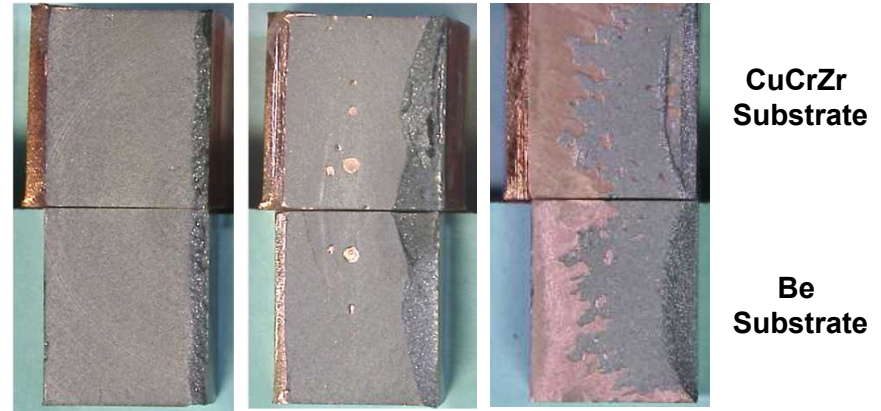


# Shear strength summary- sputter deposition



- 580C and 560C HIP joints
  - High strength bonds
  - Little sample-to-sample variation
- 540C HIP joints
  - Some reduction in strength
- Bonding
  - 580, 560C: little or no debonding at Cu/CuCrZr interface
  - 540C: Clear evidence of debonding at Cu interface

Increasing degree of incomplete bonding



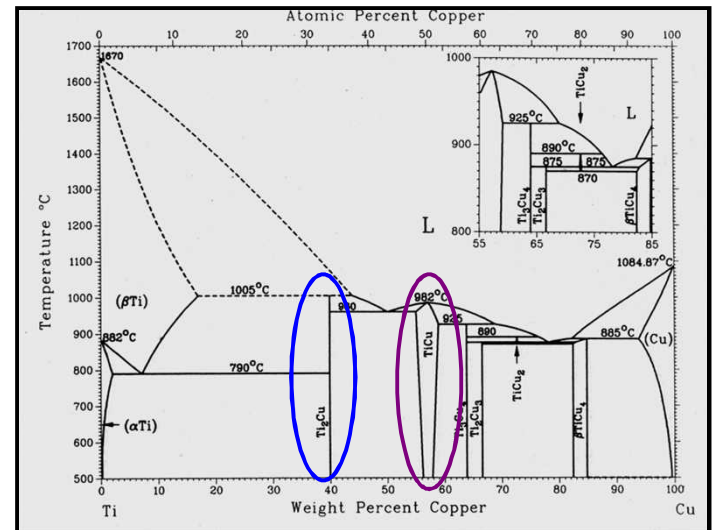
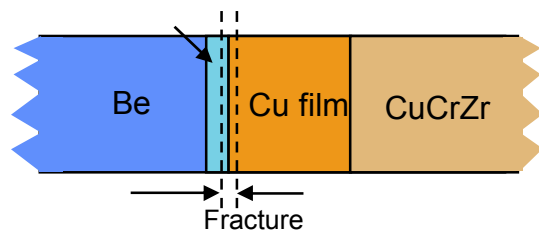
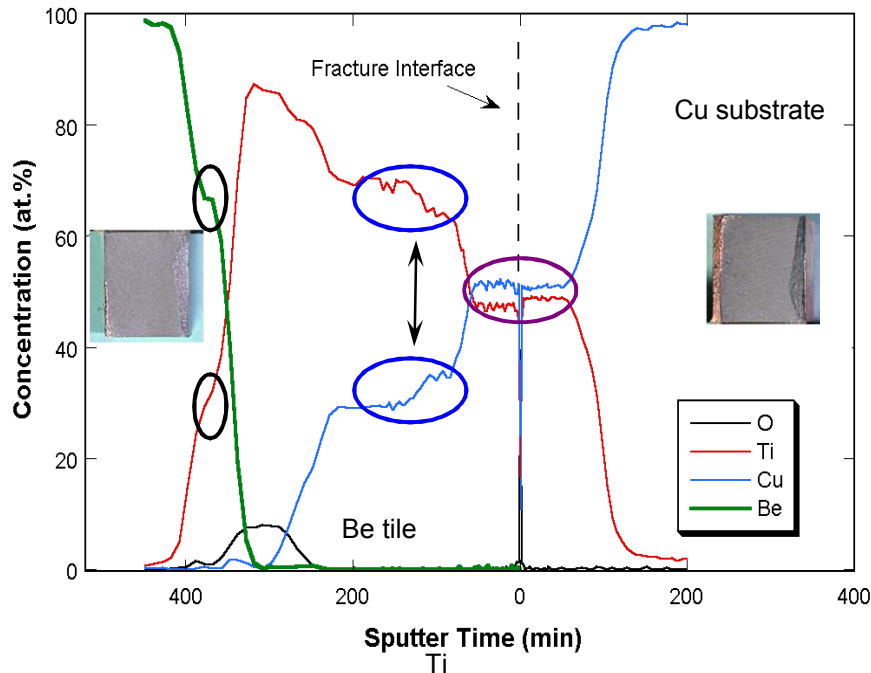
#1, 580C  
173 MPa

#4, 560C  
203 MPa

#7, 540C  
156 MPa

Bond #	Be Surface Prep.	Diffusion Barrier	Compliant Layer	HIP Temp (C)	Avg. Shear Strength (MPa)
1	(ga)(s)	2μm Ti	25μm Cu	580	173
2	(ga)(ns)	2μm Ti	25μm Cu	580	179
3	(ga)(s)	2μm Ti	3μm Cu-50μm Cu(f)	580	197
4	(ga)(s)	2μm Ti	25μm Cu	560	203
5	(ga)(s)	2μm Ti	3μm Cu-50μm Cu(f)	560	185
6	(pl)(s)	2μm Ti	25μm Cu	560	165
7	(ga)(s)	2μm Ti	25μm Cu	540	156
8	(pl)(s)	2μm Ti	25μm Cu	540	145

# Auger depth profiling of fracture surface



- Fracture is coincident with 50 at% Cu-50 at.% Ti
- Plateaus correspond to specific intermetallic compounds identifiable in the Ti/Cu phase diagram:
  - fracture surface: TiCu ( $\zeta$ -phase)
  - $\text{Ti}_3\text{Cu}$  ( $\eta$ -phase),
- Intermetallic regions most well defined for 580C HIP bonds
- Some evidence of the formation of a Be-Ti intermetallic

# Metallization/bonding schemes- E-beam evaporation

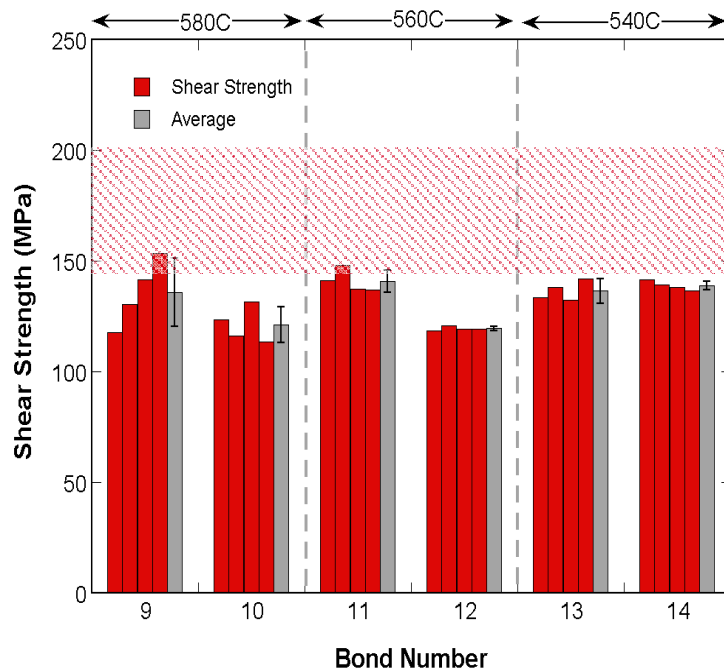
Be Surface Prep.	Metallization Process	Diffusion Barrier	Compliant Layer	CuCrZr Surface Preparation	HIP Temp (C)
(ga)(ns)	E-beam Evap.	2 $\mu$ m Ti	3 $\mu$ m Cu-50 $\mu$ m Cu(f)	Chem. Polish	580
(pl)(ns)	E-beam Evap.	2 $\mu$ m Ti	3 $\mu$ m Cu-50 $\mu$ m Cu(f)	Chem. Polish	580
(ga)(ns)	E-beam Evap.	2 $\mu$ m Ti	3 $\mu$ m Cu-50 $\mu$ m Cu(f)	Chem. Polish	560
(pl)(ns)	E-beam Evap.	2 $\mu$ m Ti	3 $\mu$ m Cu-50 $\mu$ m Cu(f)	Chem. Polish	560
(ga)(ns)	E-beam Evap.	2 $\mu$ m Ti	3 $\mu$ m Cu-50 $\mu$ m Cu(f)	Chem. Polish	540
(pl)(ns)	E-beam Evap.	2 $\mu$ m Ti	3 $\mu$ m Cu-50 $\mu$ m Cu(f)	Chem. Polish	540

Decreasing  
HIP  
Temperature

(ga): grit abraded, chem cleaned (f): foil  
(ns): no sputter clean (pl): lapped

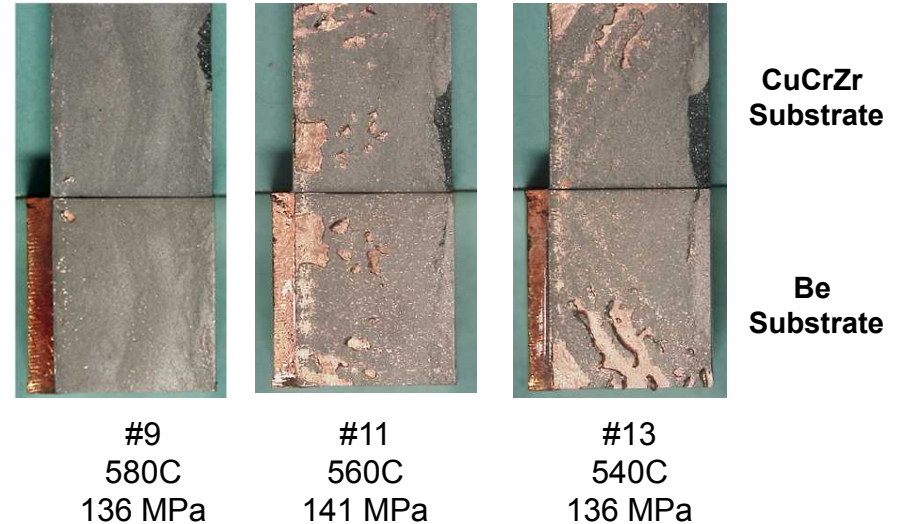
- E-beam evaporation limited to thinner films, < 5  $\mu$ m  
–Foil insert used for compliance layer

# Shear strength summary- E-beam evaporation



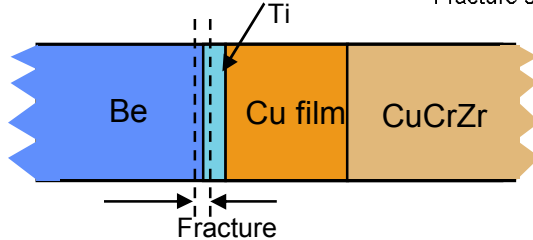
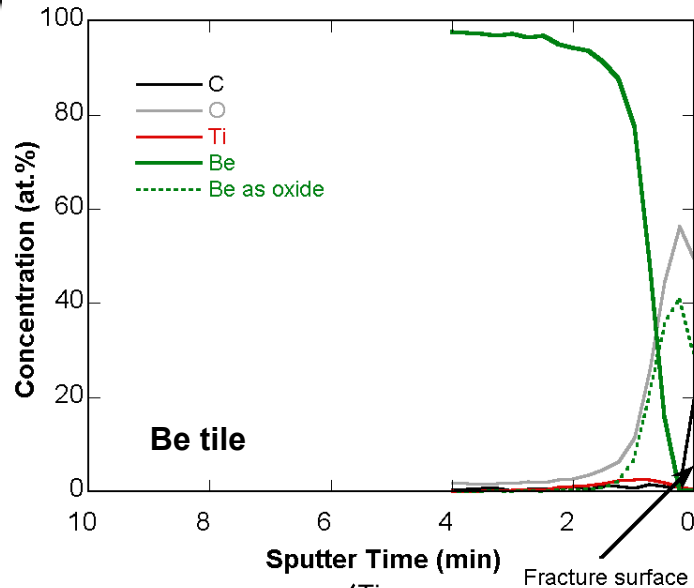
- Small scatter within any joint type
- Lower bond strength at all HIP processing temperatures compared to sputter coated Be substrates
- Cu-CuCrZr bonding compromised at 560C and 540C joining temperatures

Increasing degree of incomplete bonding

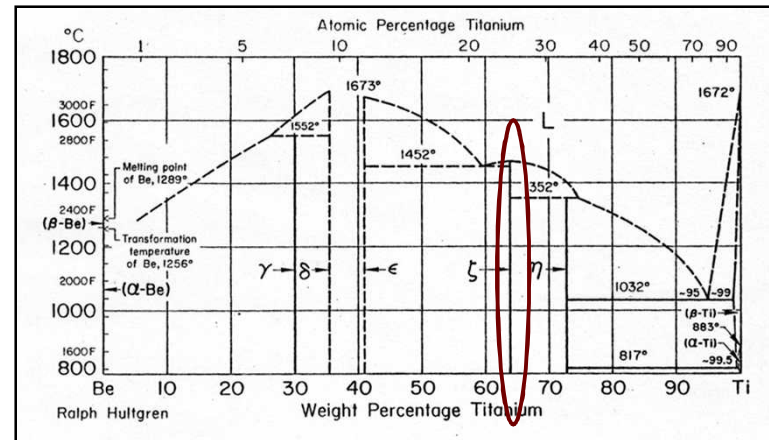
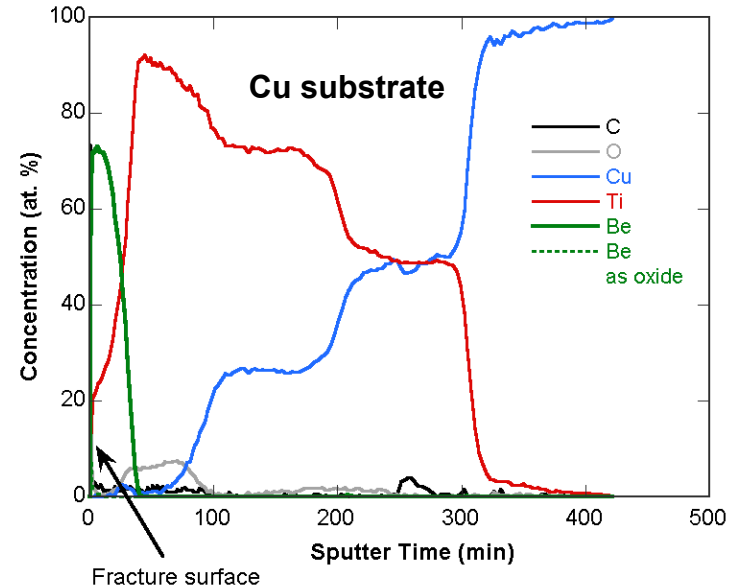


Bond #	Be Surface Prep.	Diffusion Barrier	Compliant Layer	HIP Temp (C)	Avg. Shear Strength (MPa)
9	(ga)(ns)	2μm Ti	3μm Cu-50μm Cu(f)	580	136
10	(pl)(ns)	2μm Ti	3μm Cu-50μm Cu(f)	580	121
11	(ga)(ns)	2μm Ti	3μm Cu-50μm Cu(f)	560	141
12	(pl)(ns)	2μm Ti	3μm Cu-50μm Cu(f)	560	120
13	(ga)(ns)	2μm Ti	3μm Cu-50μm Cu(f)	540	136
14	(pl)(ns)	2μm Ti	3μm Cu-50μm Cu(f)	540	140

# Auger depth profiling of fracture surface



- Same Cu/Ti intermetallics form
- A Be-Ti intermetallic exists at about 75at.% Be
- Fracture follows the interface between the Be and a Be/Ti intermetallic
- Fracture surface and depth profiling same for all HIP processing temperatures





# Summary

---

- HIP bonding schemes are yielding high quality bond joints
  - High strength
  - Small scatter
- Thin Ti metallizations are effective in acting as a barrier film between Be and Cu
- Cu interlayers successfully accommodate interface strains
- Sputter PVD metallizations
  - Failure is in the Ti/Cu intermetallic formed during the HIP cycle
  - At lowest bonding temperature some failure in the Cu/CuCrZr interface
  - Maximum bonding strength is observed for 580C and 560C HIP bonding runs:  
≈ 170 - 200 MPa shear strength
- E-beam evaporated films
  - Lower overall bond strength: 120 - 140 MPa shear strength
  - Shear failure is at the Be-Be/Ti interface

# FW Joining Technology

## CuCrZr Properties and Bonding to Stainless Steel

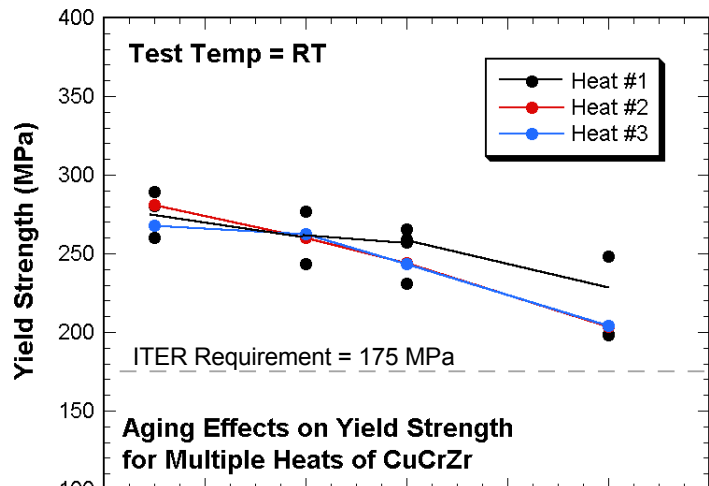
S.H. Goods<sup>1</sup>, J.D. Puskar<sup>2</sup>

*Sandia National Laboratories,*

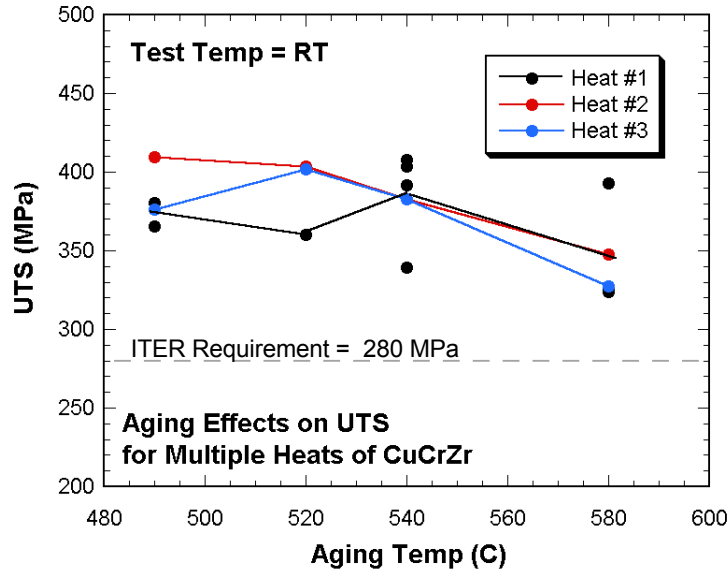
*<sup>1</sup>Livermore, CA, USA, 94550 and*

*<sup>2</sup>Albuquerque, NM, USA, 87185*

# Properties of Water Quenched CuCrZr @ RT



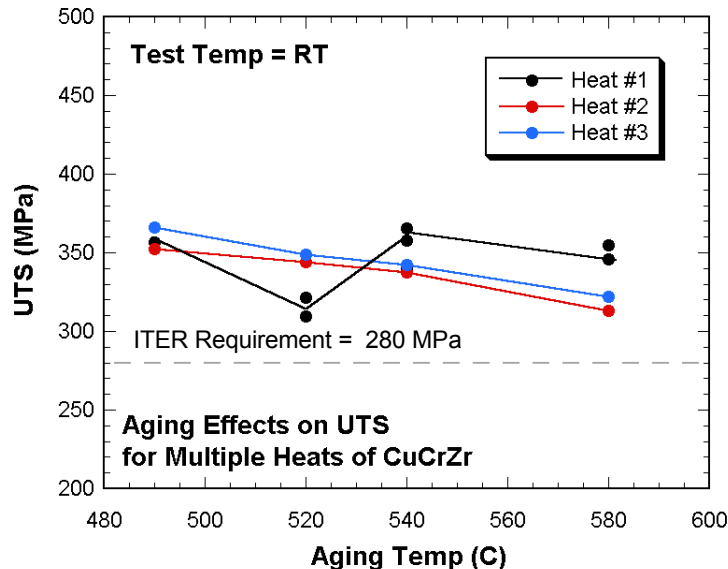
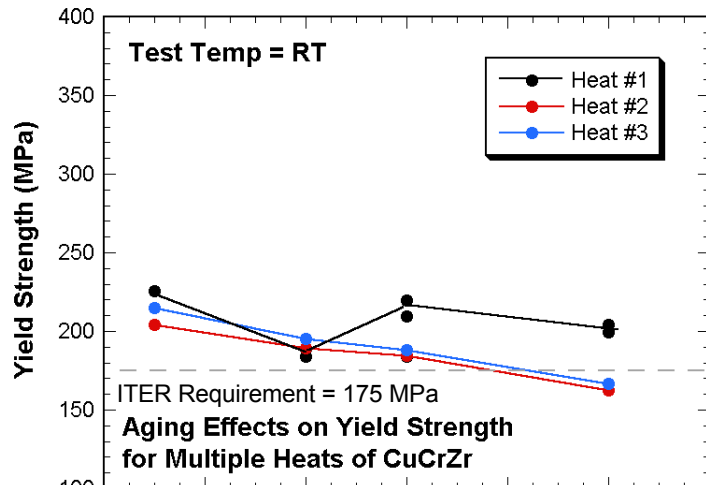
Heat	Cr	Zr	Other
#1	0.8	0.10	0.09
#2	0.9	0.07	0.05
#3	0.8	0.10	0.08
ITER	0.6-0.9	0.07-0.15	< 0.2



- Solutionized @980C/water quenched
- Aged and cooled to simulate Be/Cu HIP bonding cycle (2 hrs at indicated temperature, slow cooled)
  - Three heats meet minimum room temperature yield and UTS requirements at aging temperature  $\leq 580^{\circ}\text{C}$
  - All heats exhibit fracture strains  $\geq 20\%$



# Properties of “Slow Cooled” CuCrZr @ RT



Heat	Cr	Zr	Other
#1	0.8	0.10	0.09
#2	0.9	0.07	0.05
#3	0.8	0.10	0.08
ITER	0.6-0.9	0.07-0.15	< 0.2

- Solutionized @980C/slow cooled (as for unforced cooling in HIP furnace)
- Aged and cooled to simulate Be/Cu HIP bonding cycle (2 hrs at indicated temperature, slow cooled)
  - Two of three heats do not meet minimum room temperature yield strength requirements for 580C and are marginal for 540C aging
- CuCrZr-SS HIP bonded structures will need to be resolutionized and water quenched prior to Be-Cu bonding

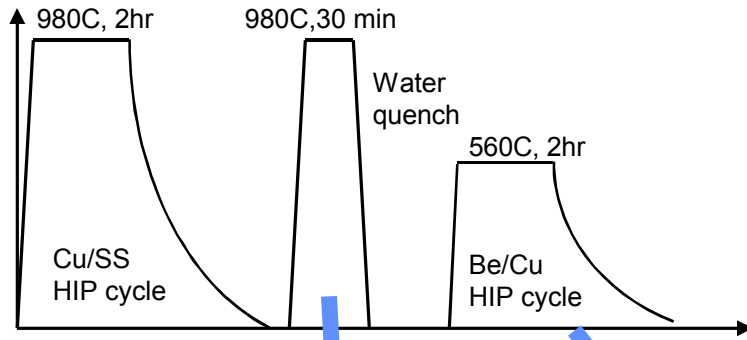
# CuCrZr-SS HIP Bonding

- Cu alloy and SS substrates
  - 5 cm diameter x 1.5 cm tall disks)
  - 316L stainless steel (solution annealed)
  - CuCrZr composition meets ITER composition specification
- Cu alloy and SS substrate preparation
  - Mechanically prepared surfaces
  - Chemically cleaned
- HIP bonding profile:
  - 980C, 100 MPa, 2 hr

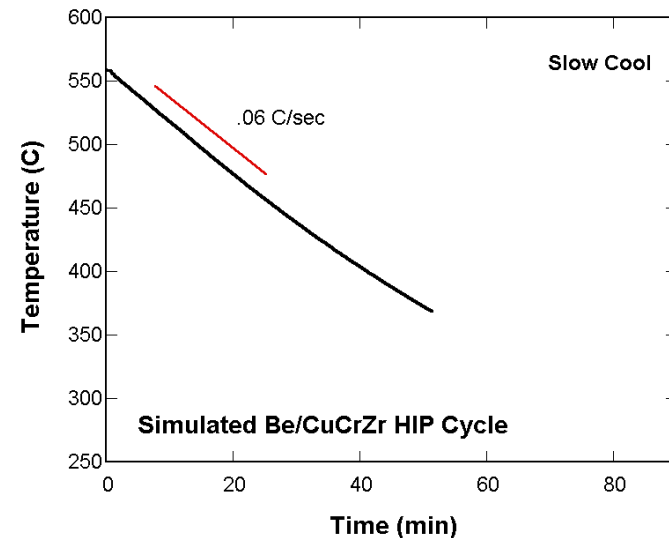
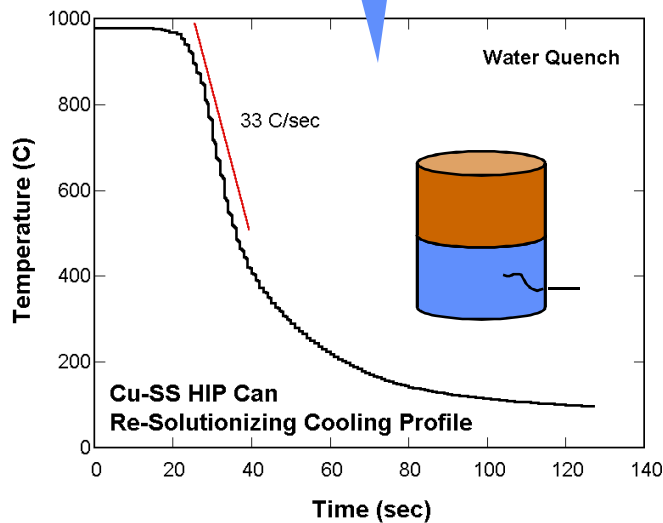


Heat	Cr	Zr	Other
#1	0.8	0.10	0.09
#2	0.9	0.07	0.05
#3	0.8	0.10	0.08
ITER	0.6-0.9	0.07-0.15	< 0.2

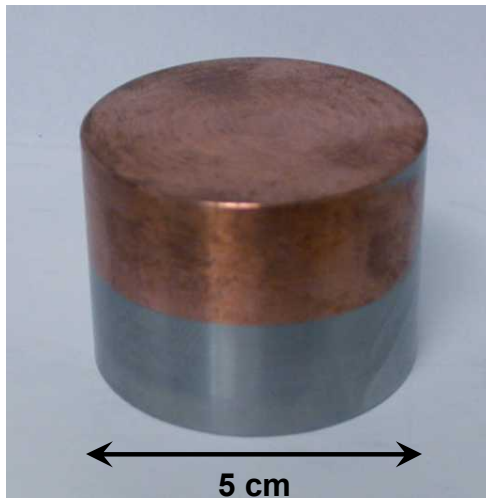
# SS-CuCrZr Additional Thermal Treatment



- Canned structure resolutionized
  - 980C/30 min, water quenched (monitored w/embedded TC in SS witness cyl.)
    - Effective cooling rated: 33C/sec
- Subjected to simulated Be/CuCrZr HIP cycle prior to extraction to test specimens:
  - 560C, 2hr, slow cool
    - Effective cooling rate: 0.06C/sec

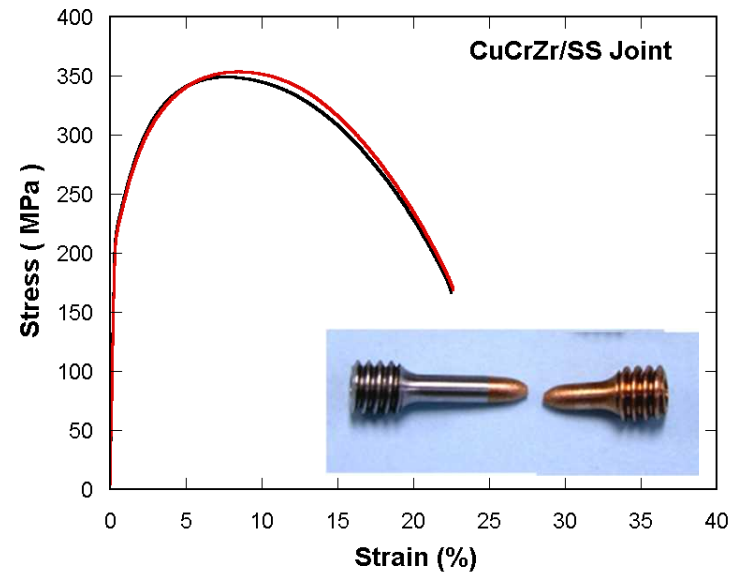
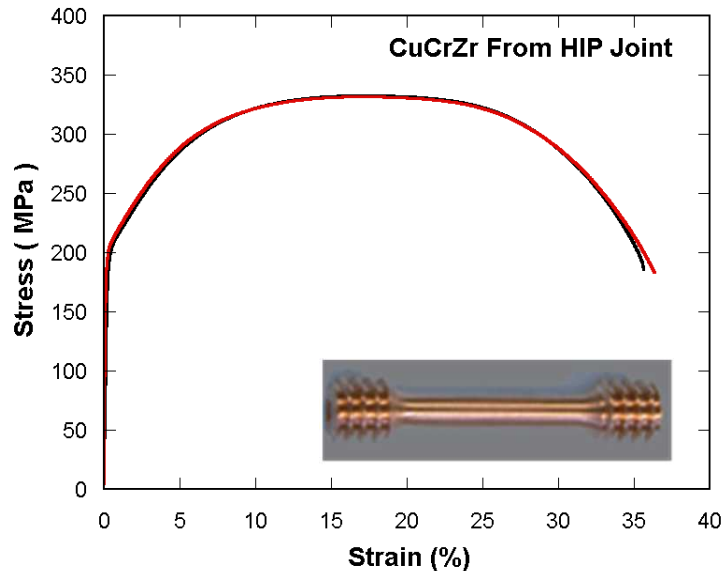


# Tensile specimen extraction



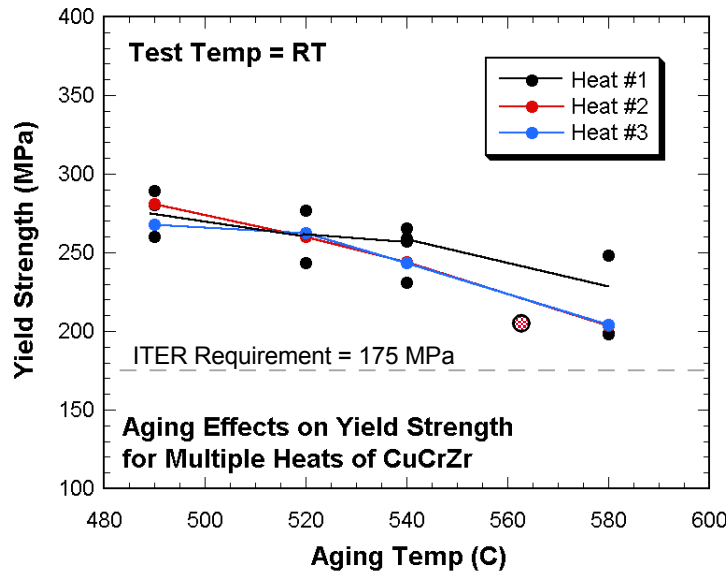
- Tensile specimens are extracted both across the bondline and parallel to the bondline fully within the copper alloy

# Mechanical Properties



- CuCrZr properties:
  - Yield Strength = 205 MPa
  - UTS = 355 MPa
- Strength of the Cu/SS joint exceeds 350 MPa
- Microscopy and compositional analysis of the bond interface are in progress

# Comparison to heat treated tensile specimens

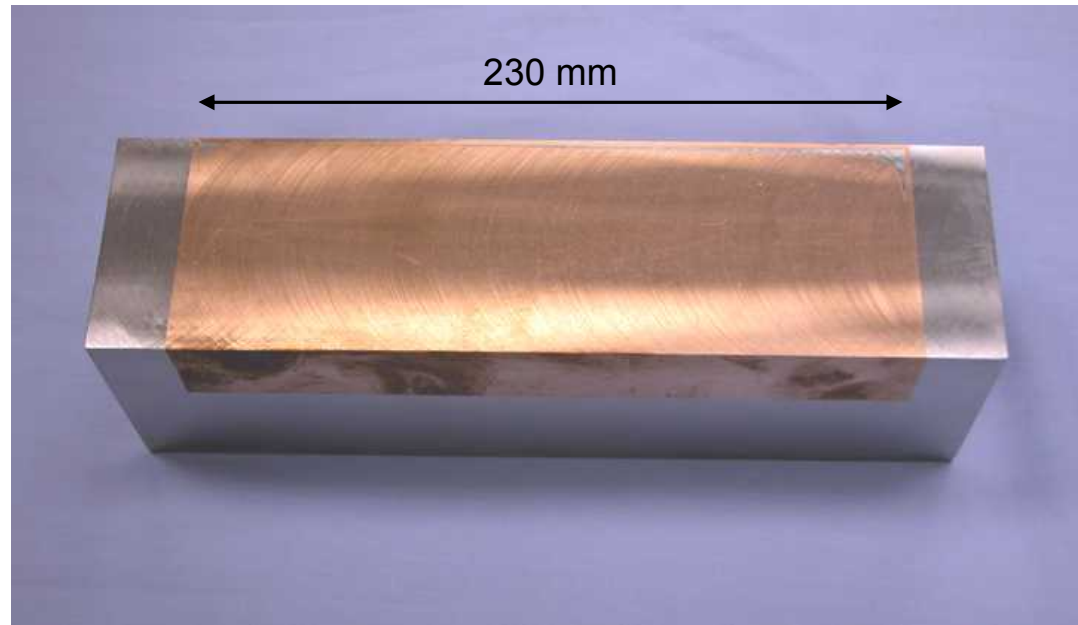


Heat	Cr	Zr	Other
#1	0.8	0.10	0.09
#2	0.9	0.07	0.05
#3	0.8	0.10	0.08
ITER	0.6-0.9	0.07-0.15	< 0.2

- CuCrZr properties after 560C heat treatment:
  - Strength exceeds ITER requirement
    - Yield Strength = 205 MPa
    - UTS = 330 MPa
- Strength after 580C HIP process will be marginal
- Strength after HIP processing at >580C will not meet minimums

# Work in progress

---



- HIP processing of FWQM-like structure
  - HIP bonded at 980C, 100 MPa, 2hrs
  - Re-solutionized and quenched
  - 560C simulated Be-CuCrZr bonding cycle
- Assess properties of interface and Cu alloy
- Patterned intentional defects for UT development

# Summary

---

- CuCrZr properties
  - Solutionizing and quenching will be necessary to meet strength requirements after Be tile bonding
  - Unlikely to be much margin on strength for Be-CuCrZr HIP bonding temperature of 580C
  - CuCrZr strength will fall below 175 MPa minimum for Be-CuCrZr HIP bonding temperature >580C
- CuCrZr-SS bonding
  - Joint strength is > 350 MPa, well in excess of Cu alloy strength