# Materials and Manufacturing Challenges for Compact Heat Exchangers for Supercritical $CO_2$ Power Systems

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U.S. DEPARTMENT OF ENERGY

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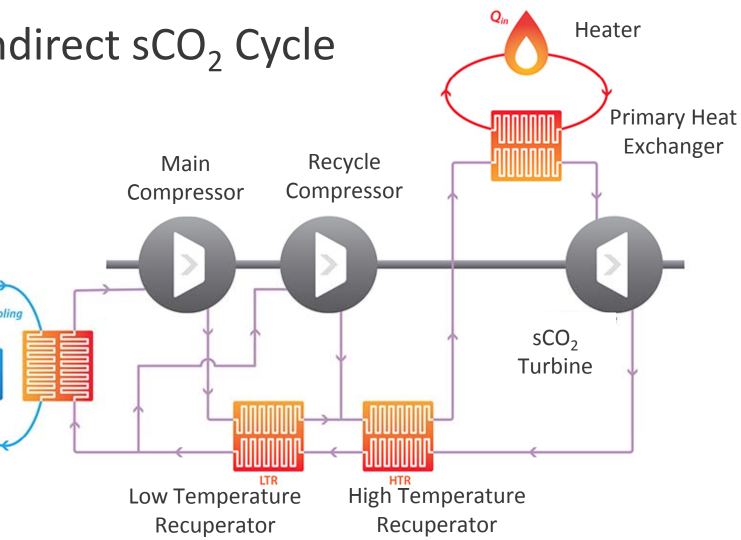
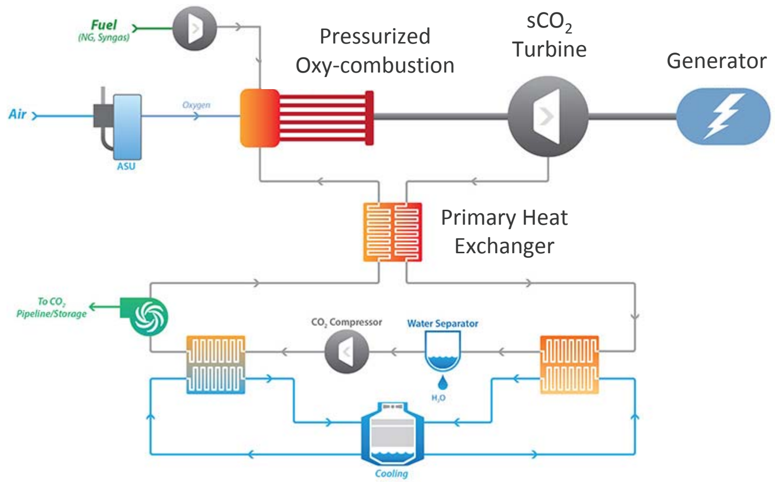
# upercritical $CO_2$ Power Cycles

ndirect $sCO_2$ Cycle




Direct $sCO_2$ Cycle




| Cycle/Component | | Inlet T (C) | Inlet P (MPa) | Outlet T (C) | Outlet P (MPa) |
|---|---|---|---|---|---|
| Indirect | Heater | 450-535 | 1-10 | 650-750 | 1-10 |
| | Turbine | 650-750 | 20-30 | 550-650 | 8-10 |
| | HX | 550-650 | 8-10 | 100-200 | 8-10 |
| Direct | Combustor | 750 | 20-30 | 1150 | 20-30 |
| | Turbine | 1150 | 20-30 | 800 | 3-8 |
| | HX | 800 | 3-8 | 100 | 3-8 |

Essentially pure $CO_2$

$CO_2$ with combustion products including $O_2$, $H_2O$ and $SO_2$

# ompact Heat Exchangers

## Higher efficiency

- Due to much shorter heat diffusion lengths in fluid

## maller size

- Use of less materials (expensive superalloys)
- Takes less space

## Modular design

- Expandable to large power plants

# ypical Compact HX Fabrication rocess

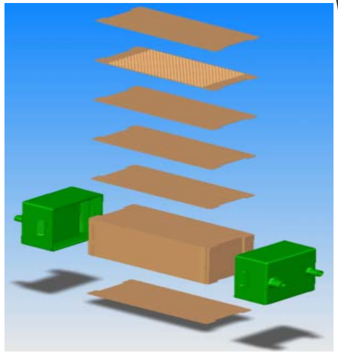
Pattern microscale flow paths into laminae using a variety of methods

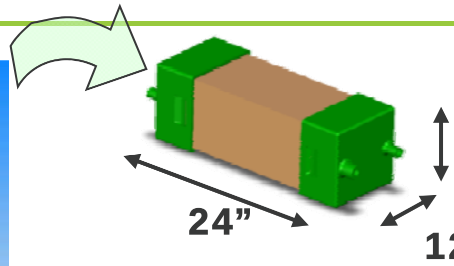
- Chemical etching
- Micromachining
- Laser cutting
- EDM

Bond these laminae using a variety of methods

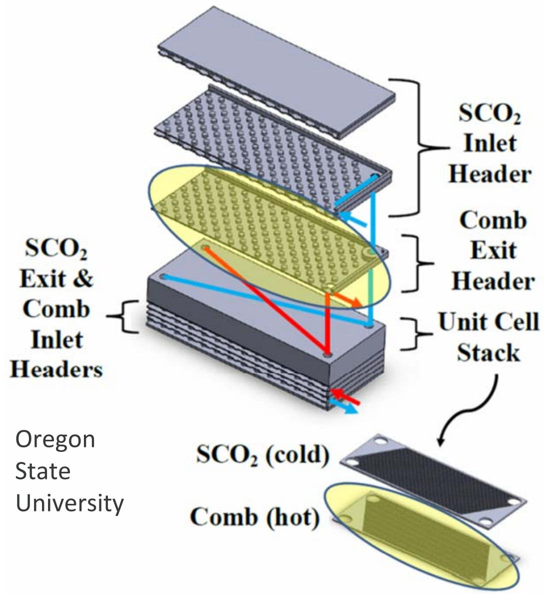
- Diffusion bonding
- Transient liquid phase (TLP) bonding
- Laser welding
- Brazing

r $sCO_2$ cycles, diffusion bonding and TLP bonding considered to be the most robust approaches






*W. Ehrfeld, V. Hessel, H. Löwe, Microreactors: New Technology for Modern Chemistry, Wiley-VCH, 2000.




Oregon State University

# onding

- Bonding is considered the "weak link" in the abrication process
- harp edges in the architecture lead to ocations of high stress concentration in the mechanical design simulations
- Ve need information on
  - The parameters for bonding process (T, P, t) of materials
  - The strength of the bond
  - Corrosion behavior of bonded regions in $sCO_2$

# aterials

High-temperature strength
High-temperature oxidation resistance

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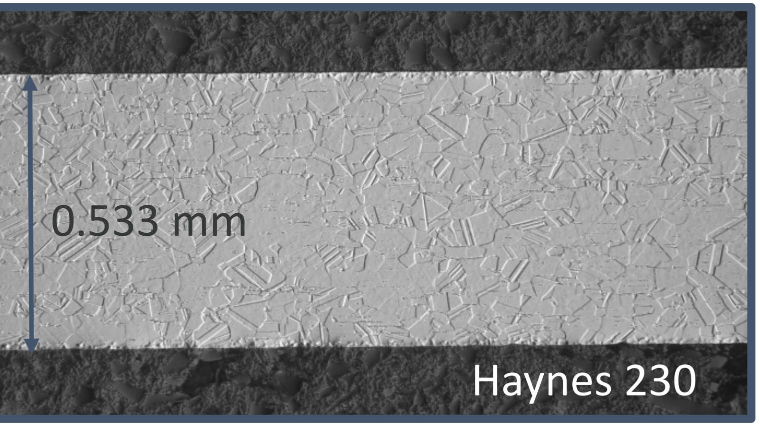
**Nominal chemical composition (weight %) of materials used in this study (Haynes 230 and Haynes 282)**

| | Ni | Cr | W | Ti | Mo | Fe | Co | Mn | Si | Al | C | B |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 57 | 22 | 14 | -- | 2 | 3* | 5* | 0.5 | 0.4 | 0.3 | 0.10 | 0.015* |
| | 57 | 19.5 | -- | 2.1 | 8.5 | 1.5* | 10 | 0.3* | 0.15* | 1.5 | 0.06 | 0.005 |

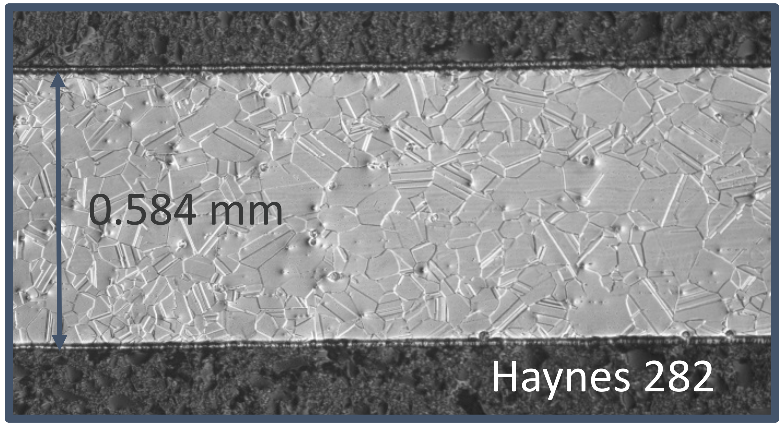
*** = maximum**

*Other materials consic for this applicatior*

Inconel 740H
Inconel 625
Inconel 617
347H Stainless stee
316 Stainless stee
304 Stainless stee
Grade 91 steel




Solid-solution strengthened
ld rolled and 1232 °C solution annealed sheet




Precipitation strengthened
1149 °C solution annealed sheet

# iffusion Bonding vs. Transient Liquid hase (TLP) Bonding



**Diffusion Bonding**

- Diffusion bonding is a solid tate process
- t requires applied high pressure at high temperature or a certain amount of time
- t involves diffusion of onstituent atoms and creep processes to close the voids present due to roughness of he faying surfaces.

**Transient Liquid Phase Bonding**

- TLP involves both solid state and liquid state reactions
- It requires less pressure than diffusion bonding
- It requires a lower melting point interlayer
- It involves isothermal melting and solidification of interlayer

# onding

heets were water-jet cut into shims

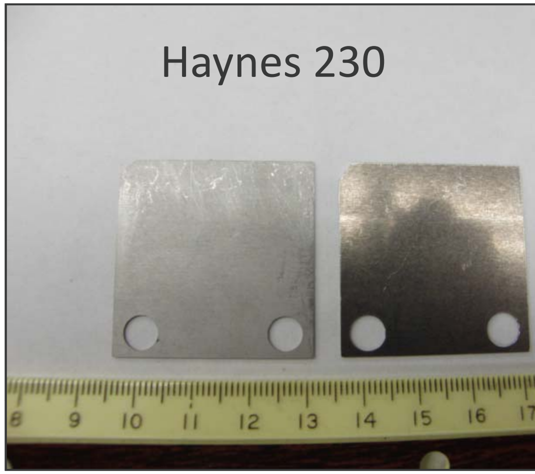
00 shims were bonded together in each stack

ll shims were reverse current etched and cleaned vith acetone

ome stacks used shims plated with electroless ickel, 2 - 4 μm thick

ome shims contained pin-fin micro-features dentical to those used in a heat exchanger

ll shims were thoroughly cleaned by hand and in an ltrasonic acetone bath for 15 minutes immediately efore bonding




# onding

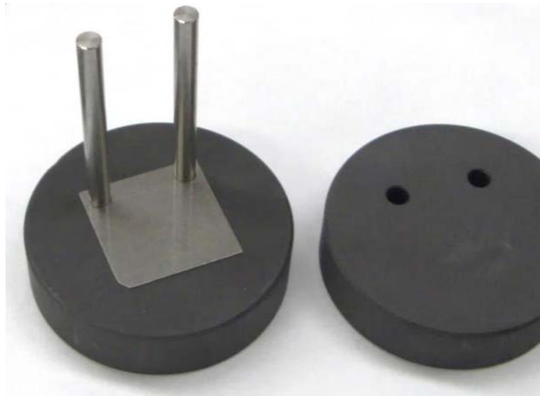
him stacks were held in a fixture during bonding nd pressure was applied only after the emperature ramped up to the desired value

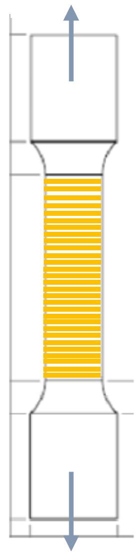
The hot press vacuum was maintained at pproximately 5 x $10^{-6}$ torr (0.0007 Pa)

150°C for 8 hours at 12.7 MPa

After bonding, each stack was machined to produce 6 tensile specimens using wire EDM and CNC lathe

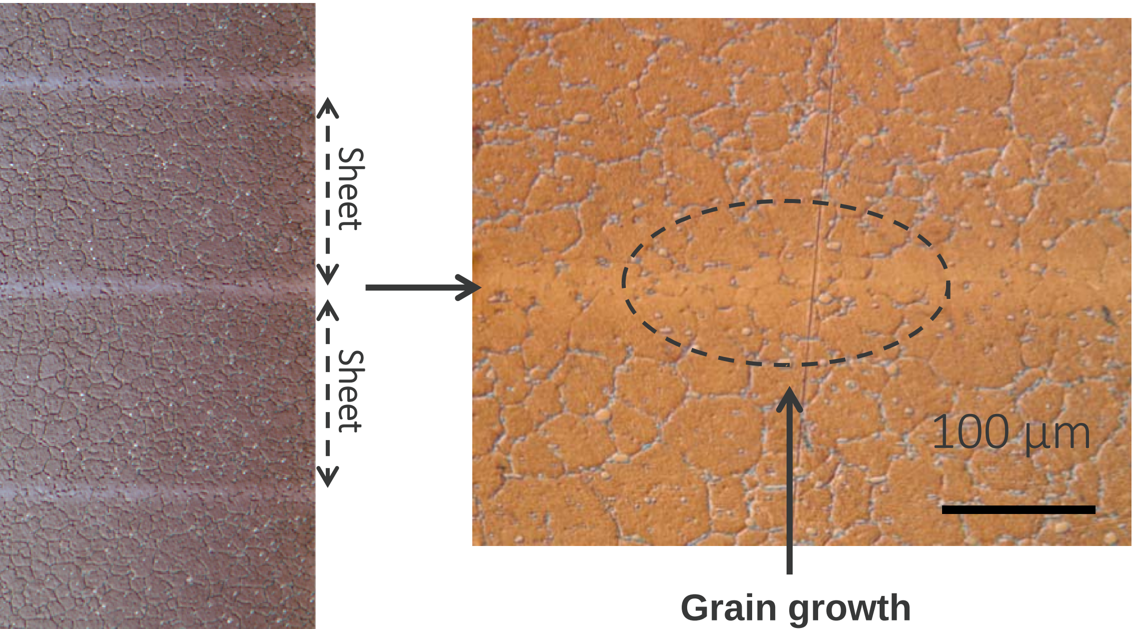
H282 without Ni plating did not bond well

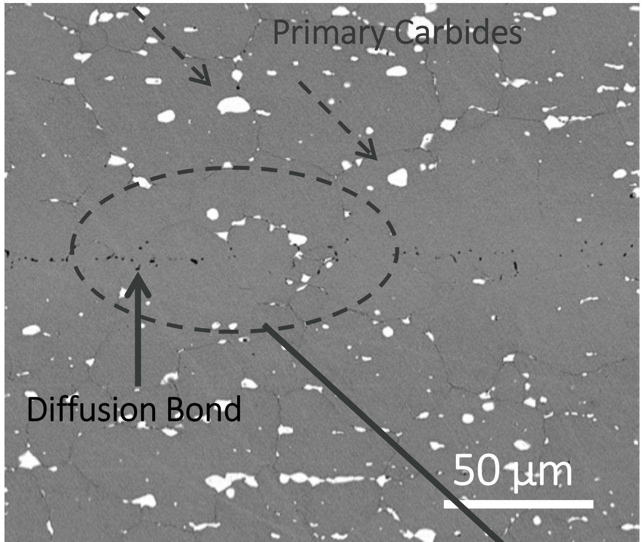


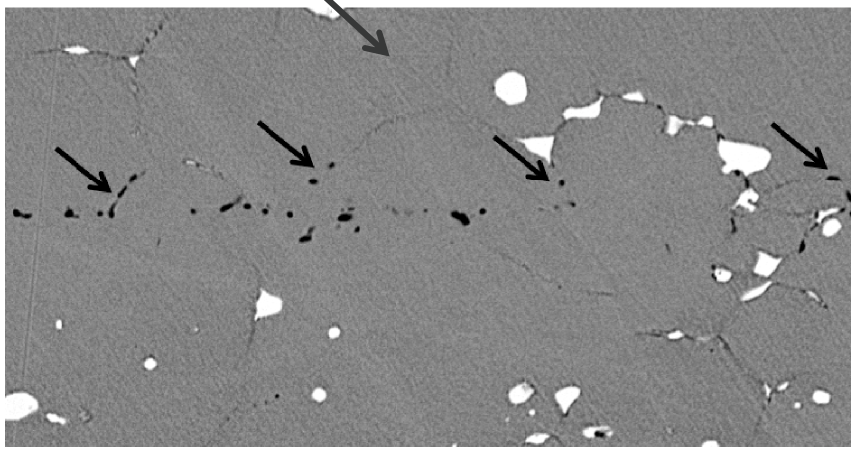


# iffusion Bonding of Alloy 230

## Microstructure



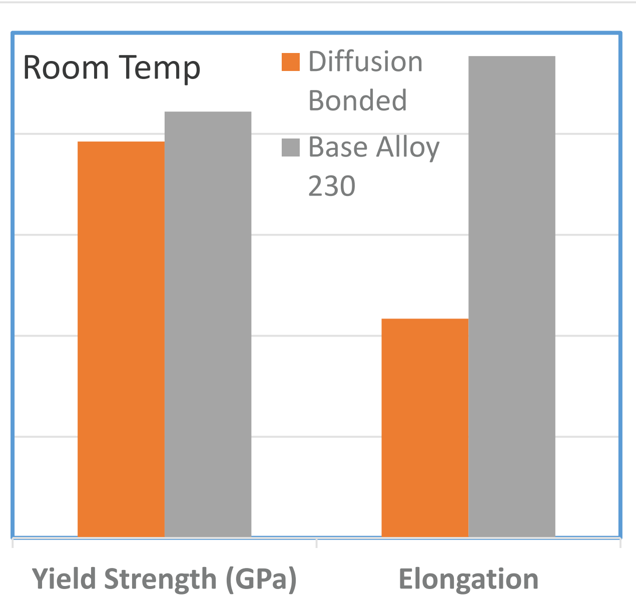



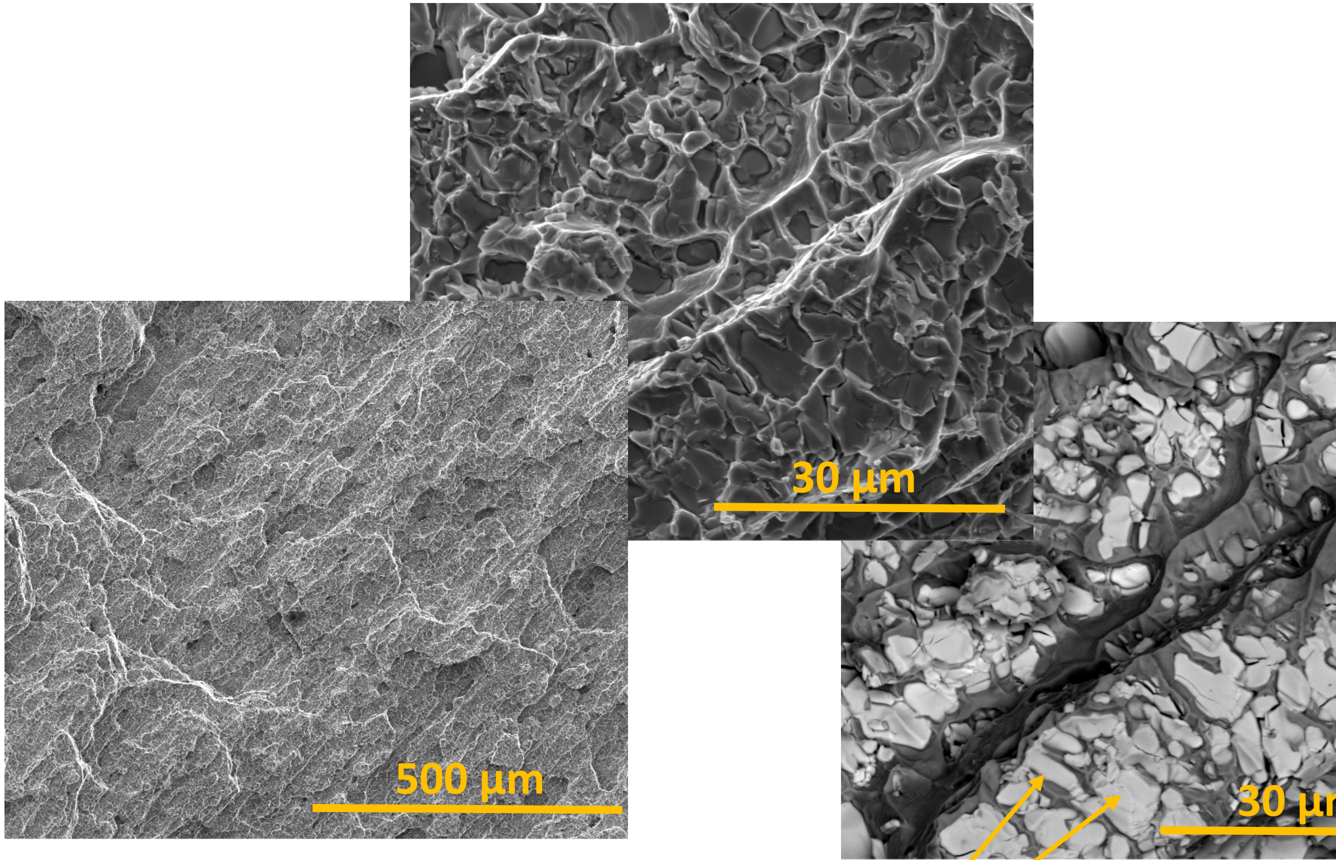
- Primary carbides form at higher temperature

tched microstructure to observe grain growth through the bond line

# iffusion Bonding of Alloy 230

## Mechanical Behavior




Fracture occurs in a ductile manner along the precipitate bands.

# P Bonding of Alloy 230

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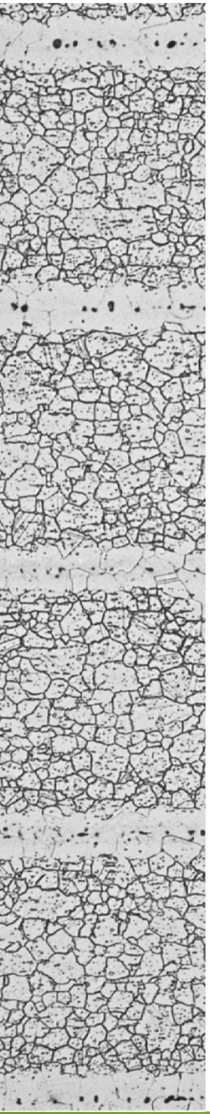
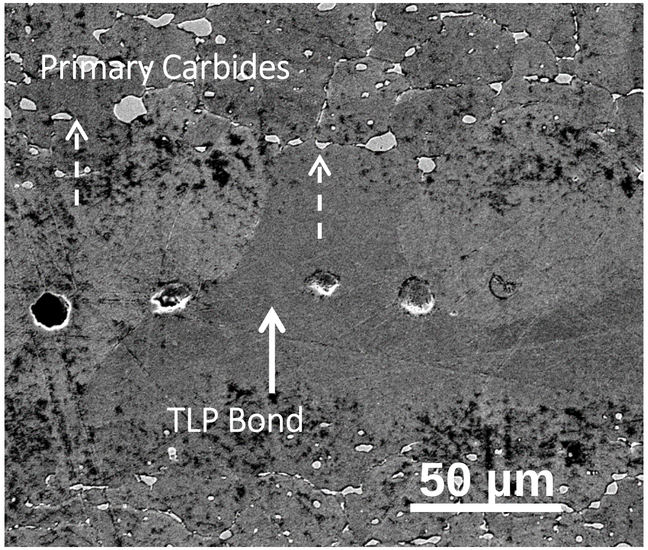
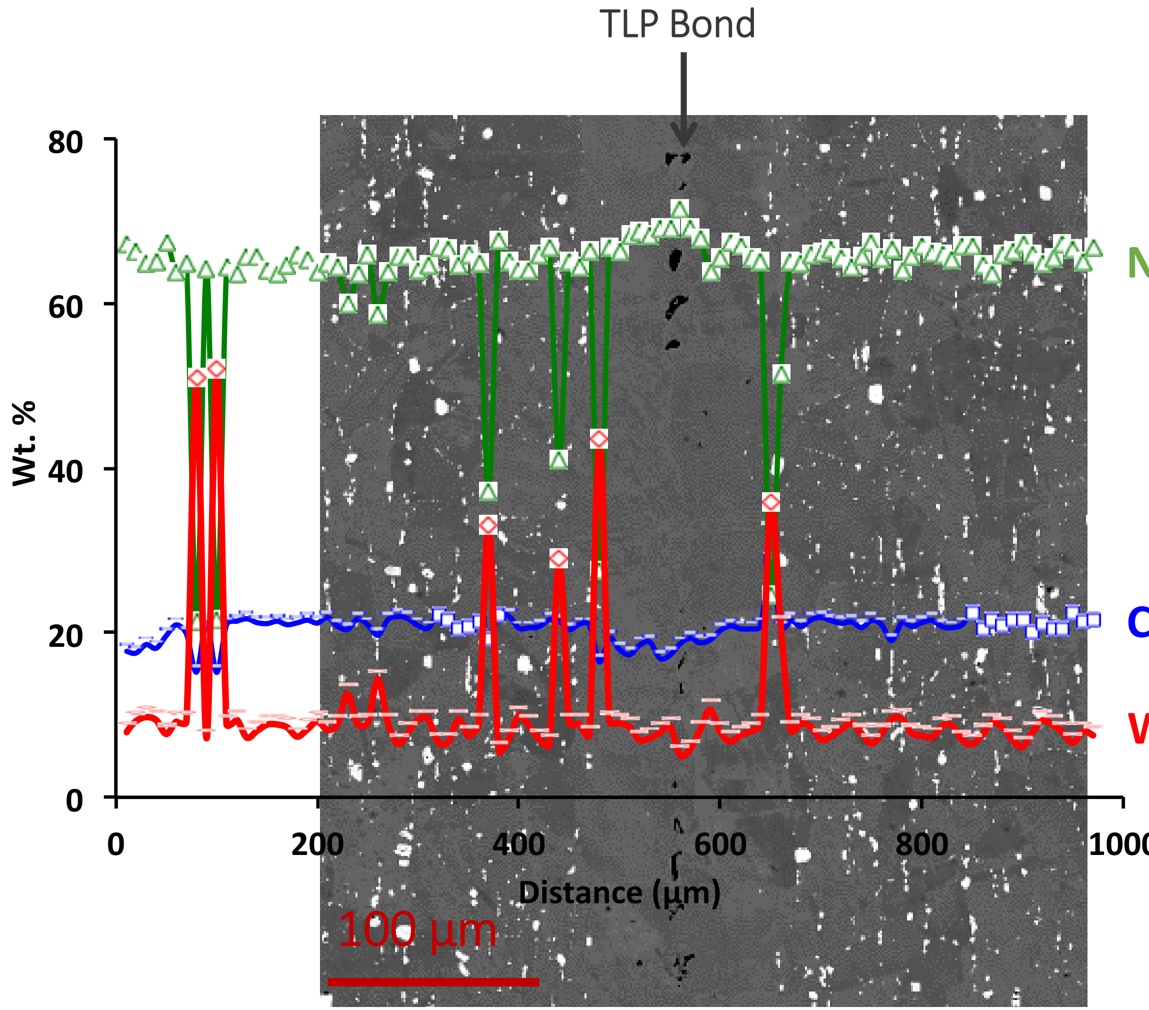


## Microstructure

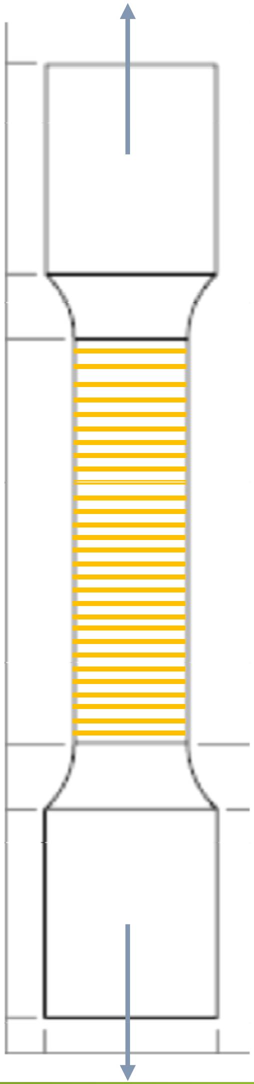



- Primary Carbides
- Increase in Ni, dip in Cr at the bond

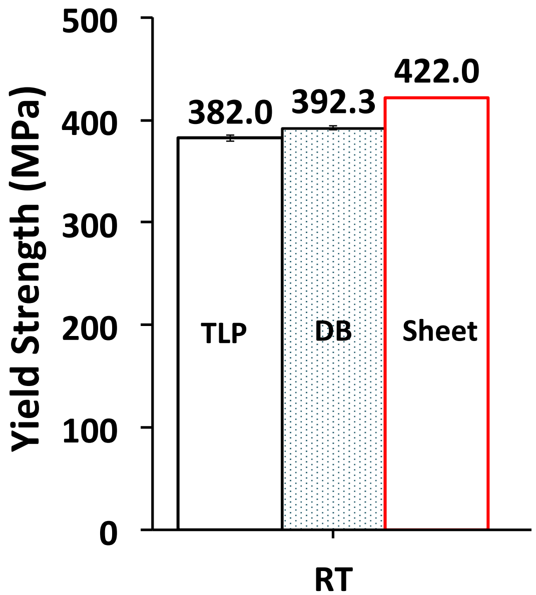


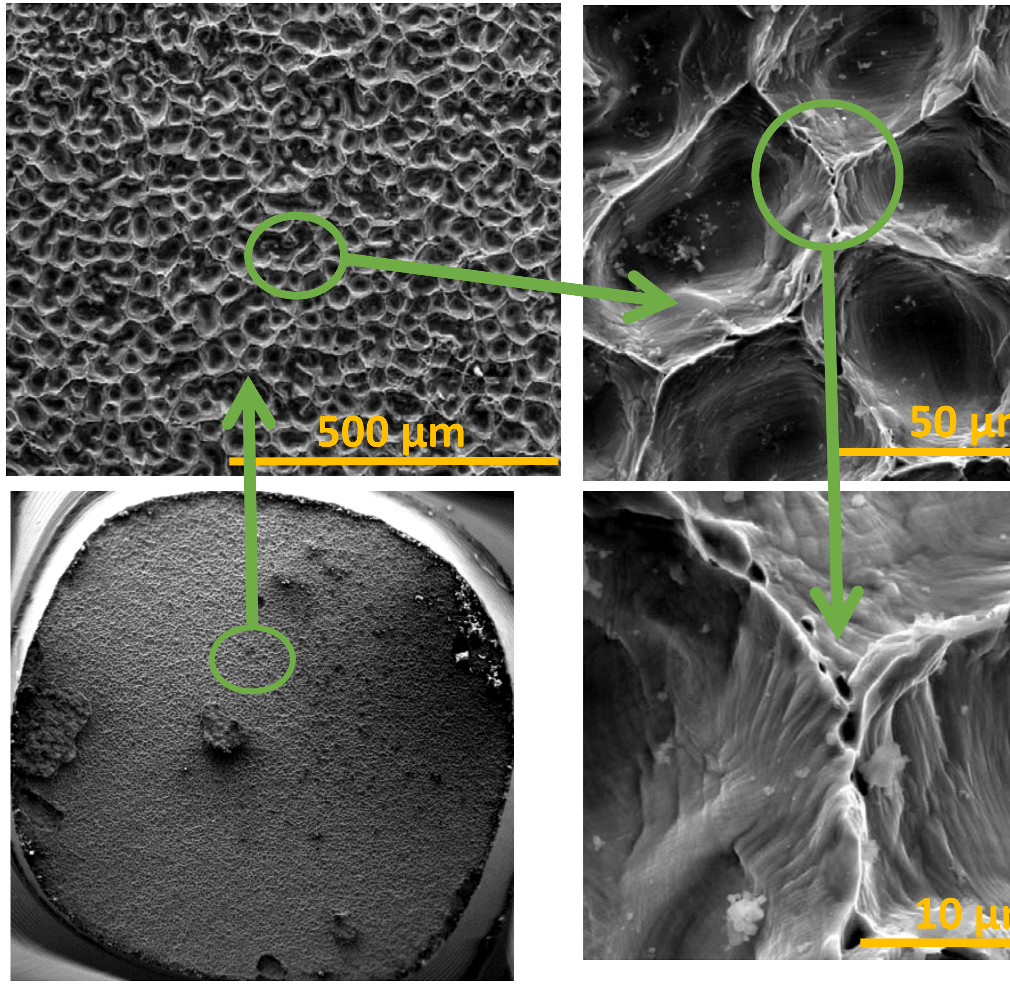
# ond Strength – TLP Bonding of Alloy 230





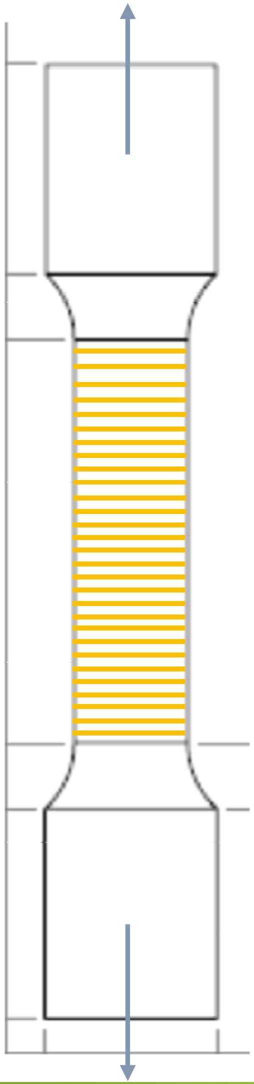
## Room Temperature Strength



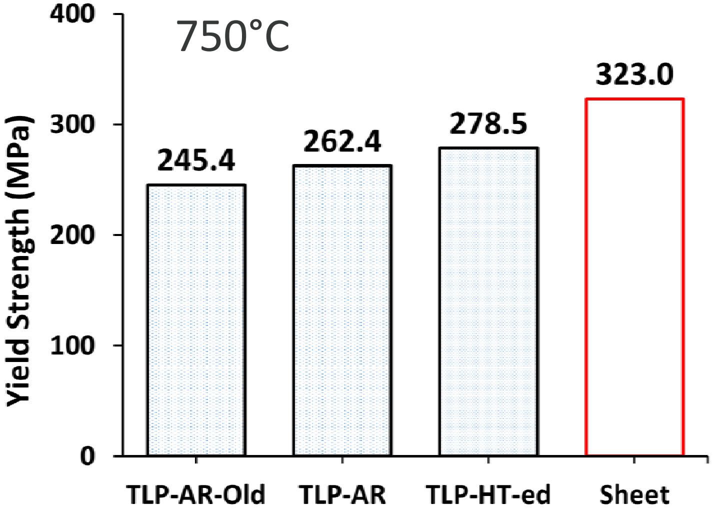




Fracture occurs in a ductile manner at the bonding layer.

# ond Strength – TLP Bonding of Alloy 230

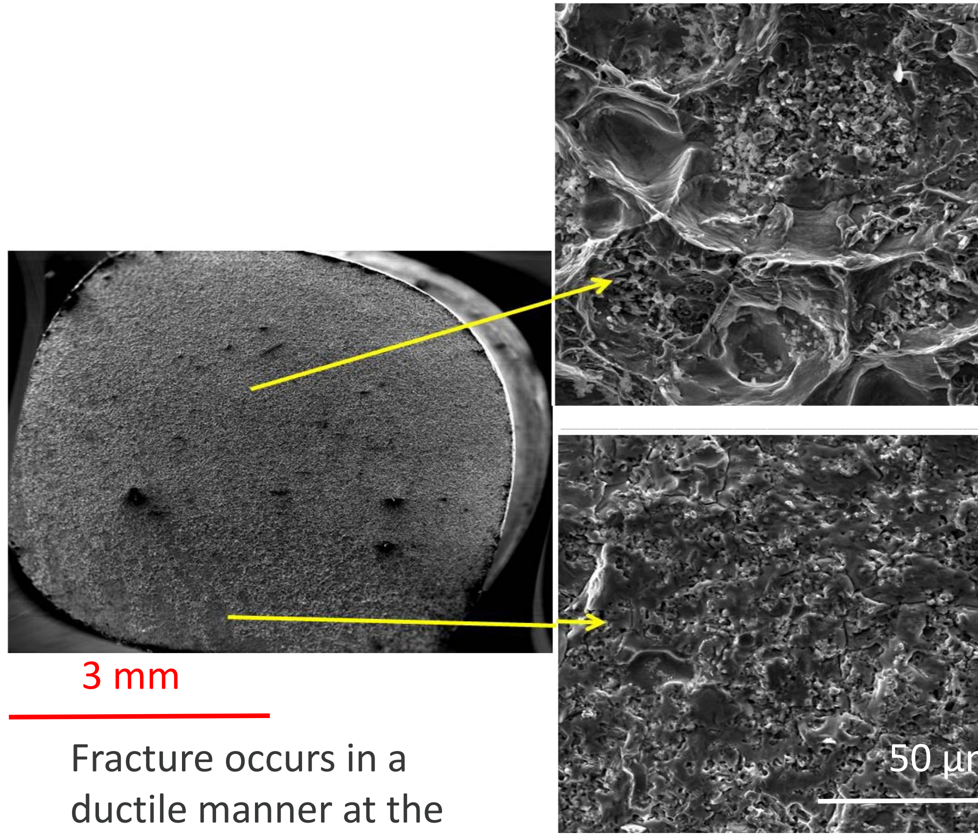


## High-Temperature Strength




**High Temperature Yield Strength**

- **TLP-AR-Old = 76%**
- **TLP-AR = 81%**
- **TLP-HT-ed = 86%**



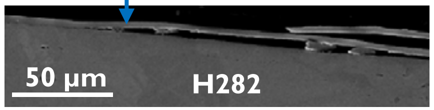

Fracture occurs in a ductile manner at the bonding layer and the sheet.

# onding Defects

## Challenges with TLP bonding of H282 – Surface oxides & Intermetallic formation

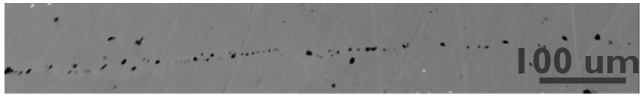
**Surface Oxides**

Ni-12P coating on the metal for TLP bonding



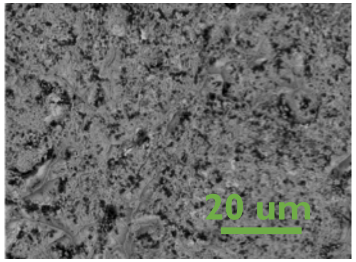

Coating does not adhere to the metal

Bondline after TLP bonding



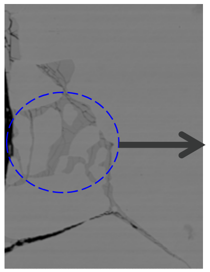

AlO particles (dark) along the bondline

Fracture Surface



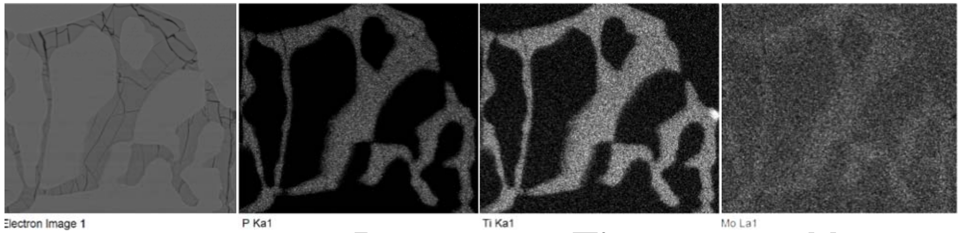

AlO particles (dark) on the fracture surface

**Intermetallic formation due to 12 wt. % P**



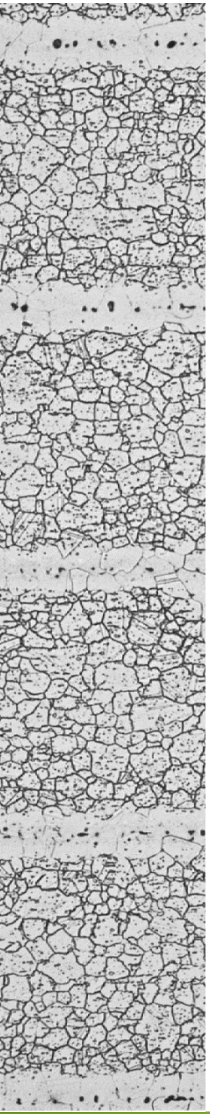
50 um

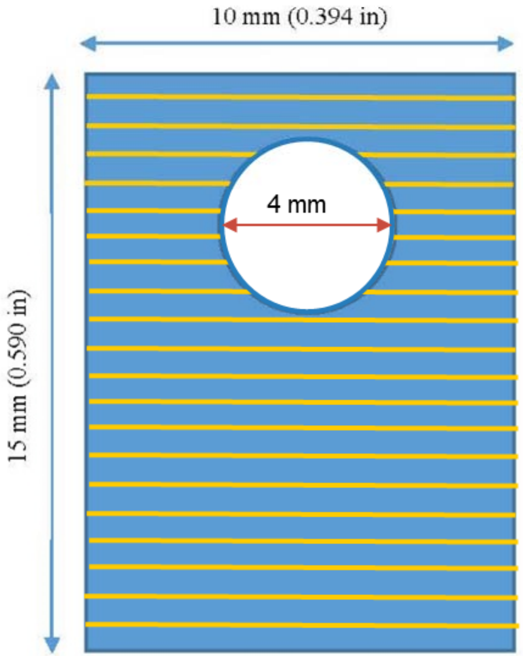
EDS maps in the vicinity of the bondline

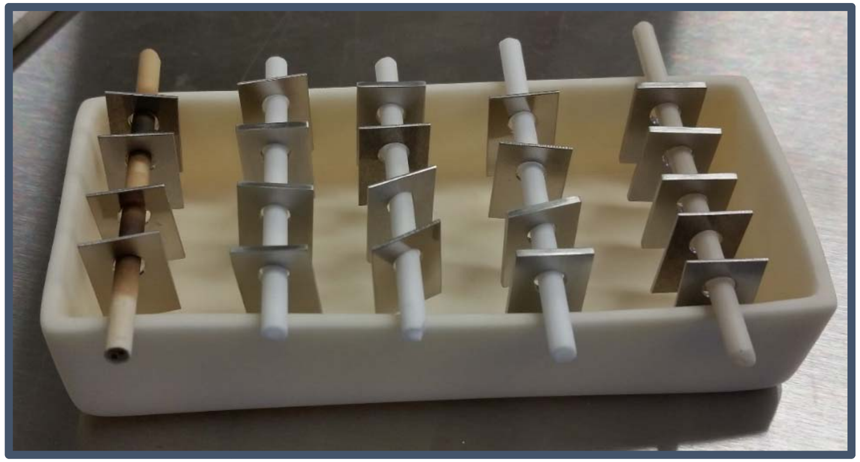



P Ti Mo

# xidation of Bonded Regions





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<u>Characterizatio</u>

Mass Change

XRD

SEM

Gas: 1 bar $CO_2$ (99.999% purity)

Gas flow rate: 0.032 kg/h

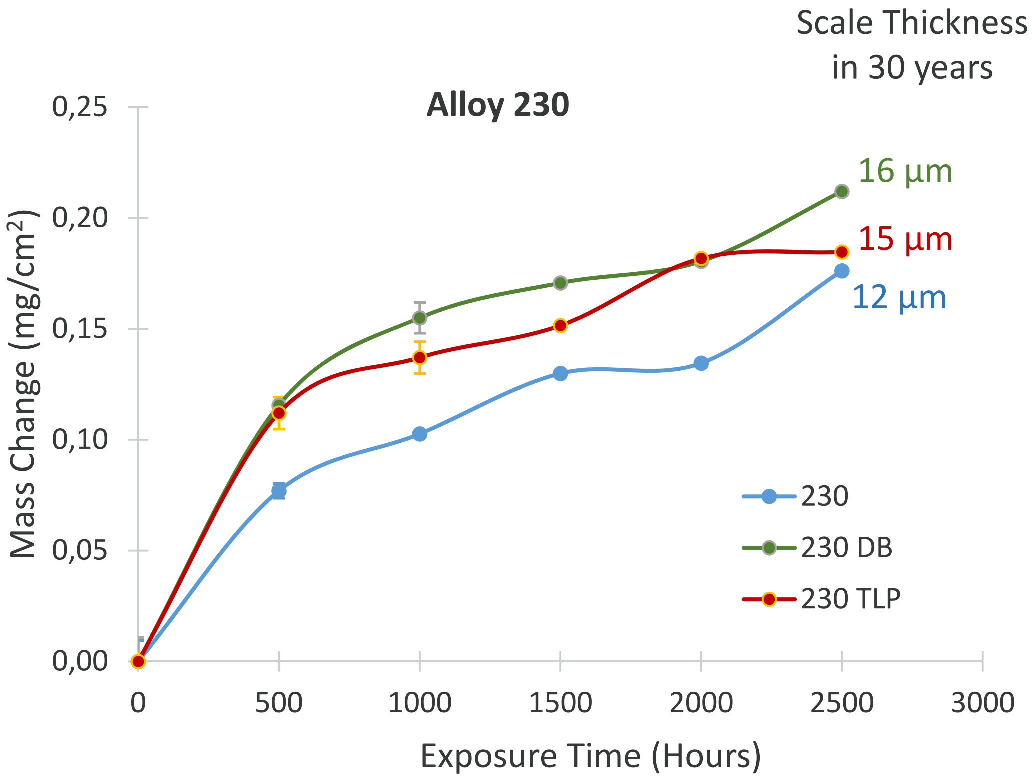
Temperature: 700°C

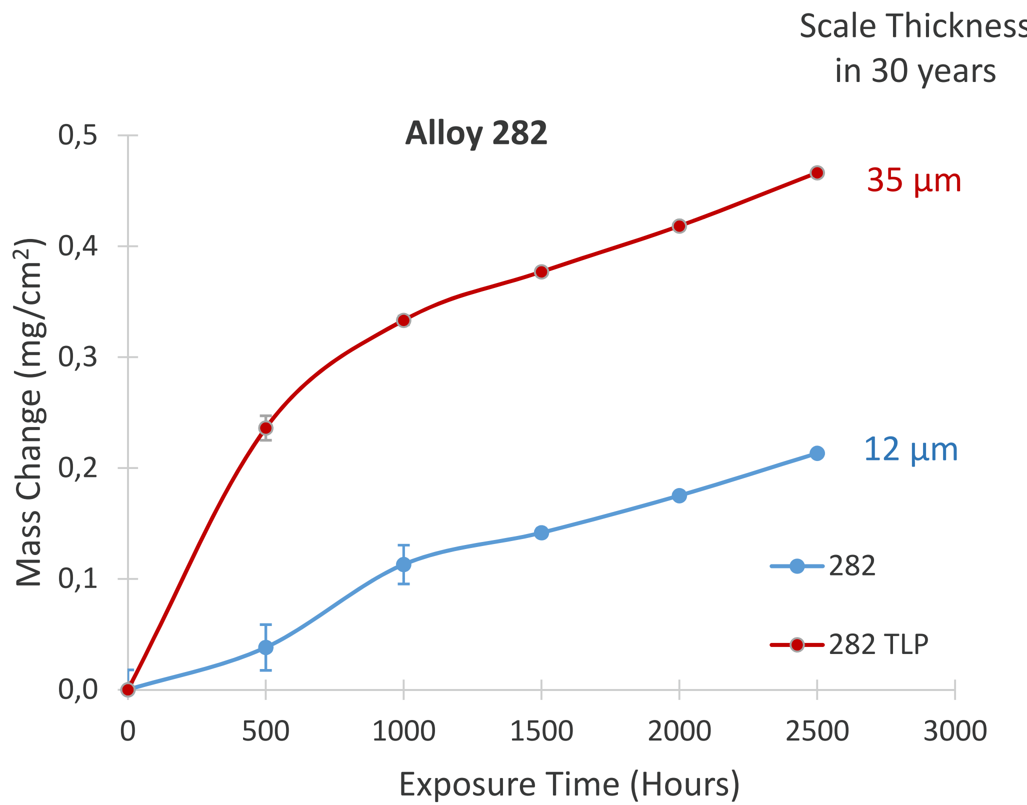
Duration: 2500 h

24 h purging with $CO_2$ before heating

# xidation of Bonded Regions

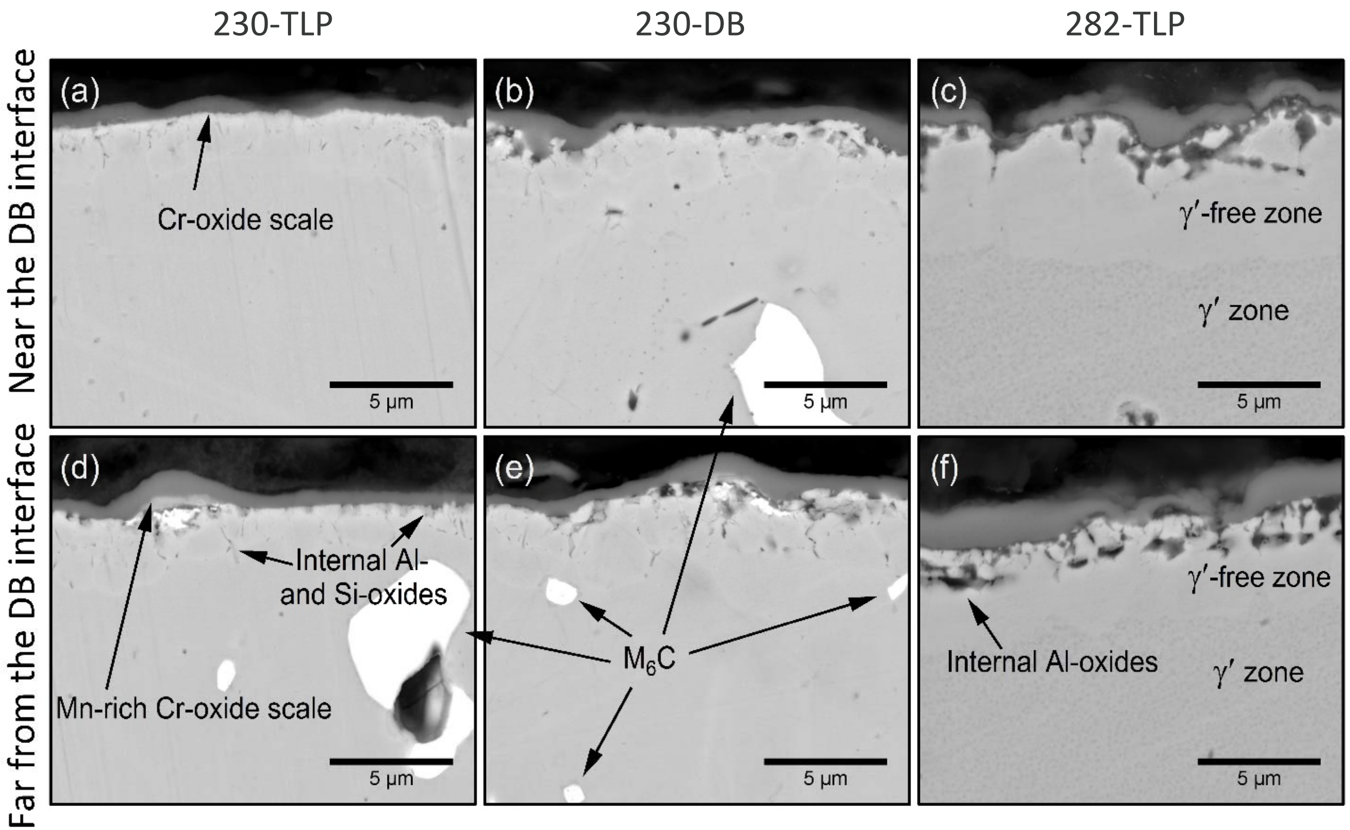
**bar $CO_2$ at 700°C**



# xidation of Bonded Regions

**bar $CO_2$ at 700°C**



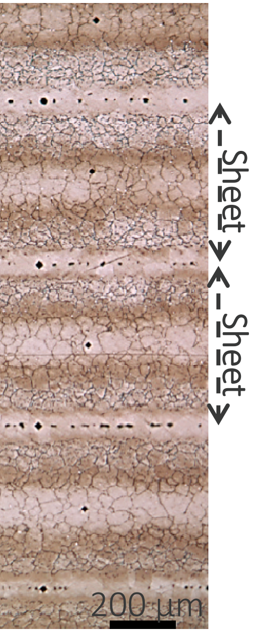

No significant difference between bond regions and away from bond regions

More internal oxidation in H282, resulting from higher Al and Ti levels

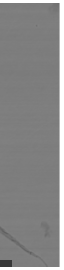
γ′ loss in H282 below the internal oxidation layer

Back-Scattered Electron Images

# ummary

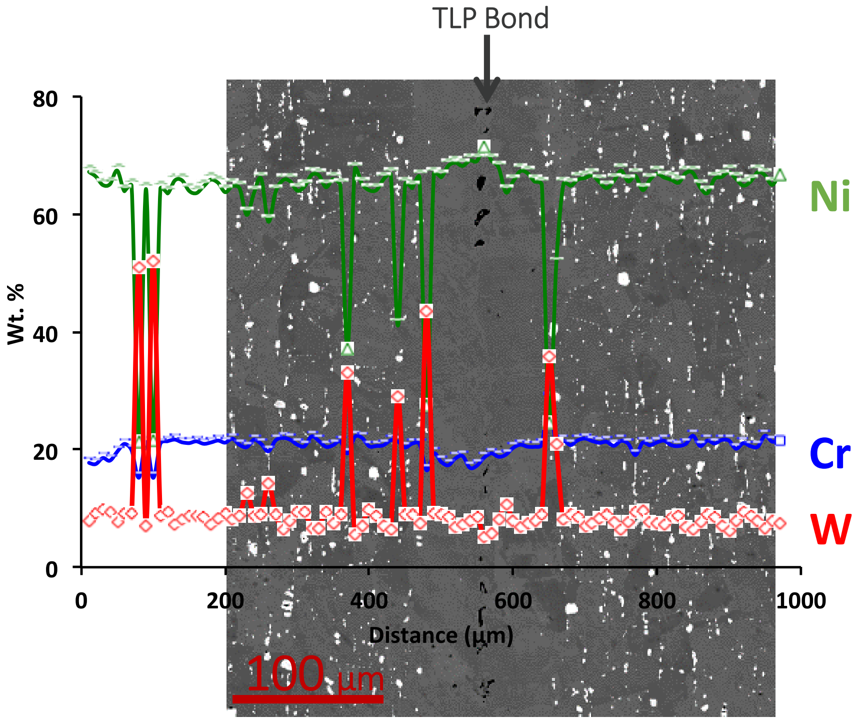



ids at bondline

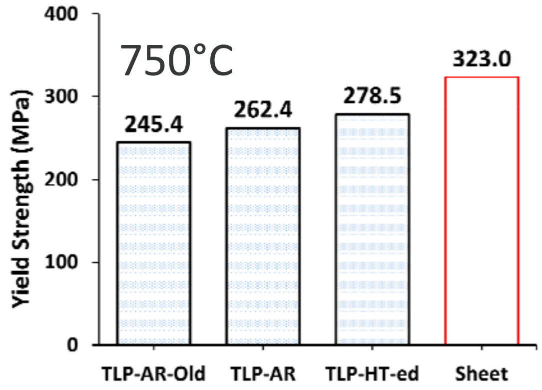


Intermetallic formation

m




Composition of TLP bonded regions were similar to the base material

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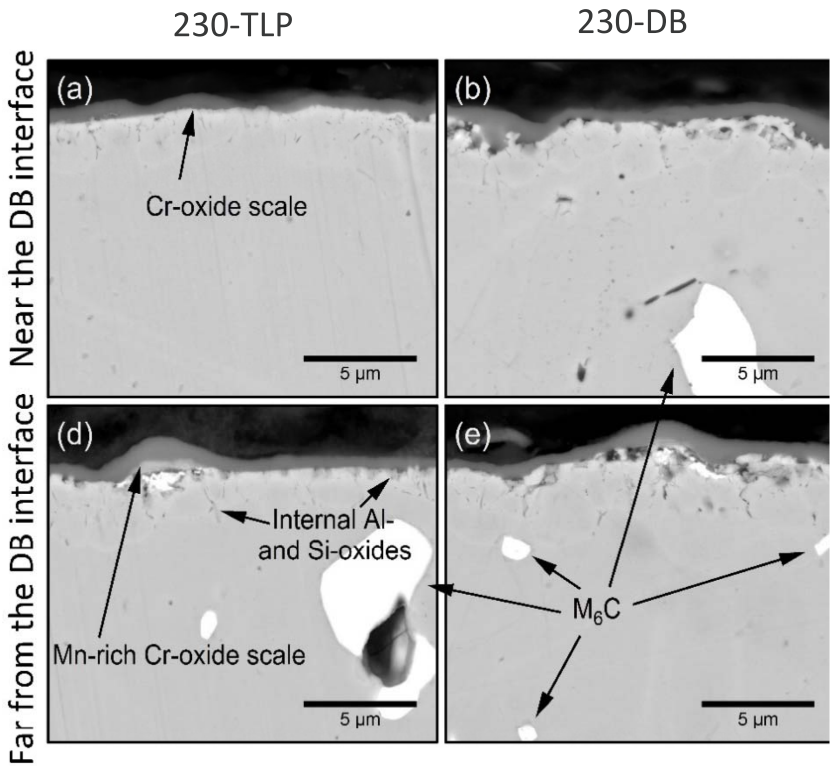
Both DB and TLP bonded stacks exhibited good strength at high and room temperature




No signific difference oxidation behavior i $CO_2$ at 700 between b regions an away from bond regio