Effect of Post-Bond Heat Treatment on the Diffusion Bonding Properties of Alloy 800H with Ni-Foil Interlayer

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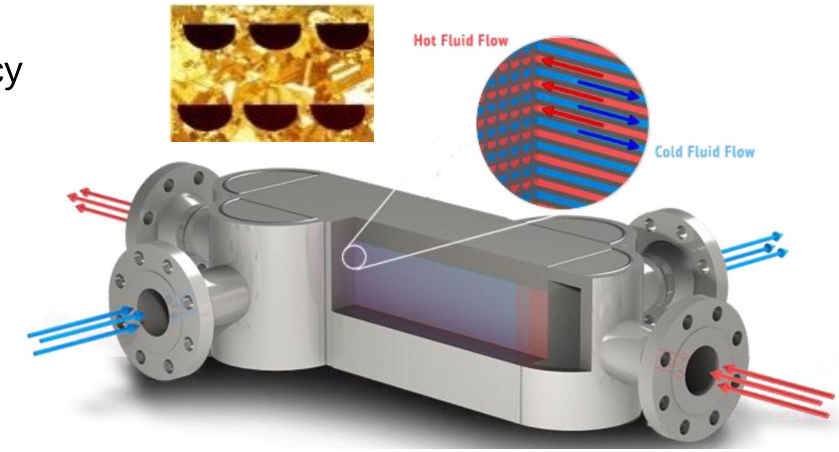
Contents

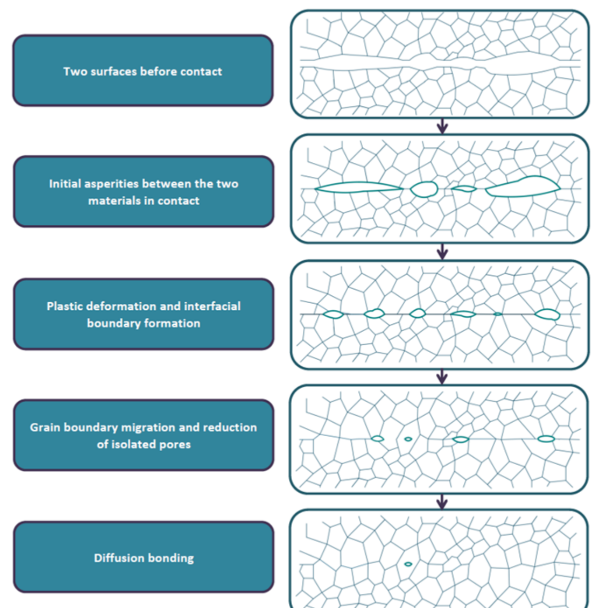
S.R. Aakre et al., S-CO_2 power cycles symposium (2018)
J.K. Min et al., Heat Mass Transfer (2009)

❑ Printed-circuit heat exchanger (PCHE)

- Micro-channel heat exchanger
  - Compact & extended surface for high efficiency
- Possible applications
  - Nuclear, fossil, solar power using S-CO_2
- Photo-chemical etching of plates
- Stacking + Diffusion bonding
- **Key component**
  - **Long-term integrity of diffusion-bonded joints is crucial**



HE type	Maximum pressure (bar)	Maximum temperature (°C)	Effectiveness
PHE	40	400	>90
PFHE	120	650	>90
Spiral	30	400	
Flat tube and fin	200	200	
Tubular (profile)	31	750	<85
Tubular (mini)	100	730	
PCHE	500	1000	>97
Ceramic	4	1300	



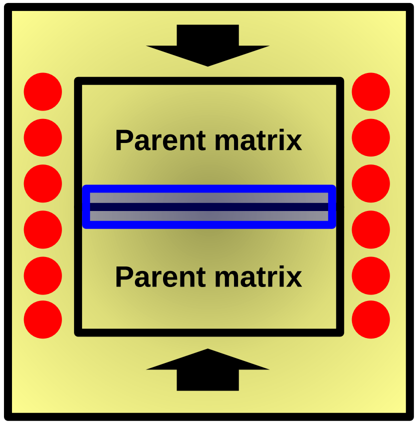
Background – DB

Aim of DB process

- Similar to the parent matrix
- Grain boundary migration across the bond-line
- Avoid excessive deformation of joint piece

Key parameters of DB process

- Surface condition
- Temperature, external pressure, time, environment



Before diffusion bonding

Surface treatment
- **Surface roughness**
  - ▷ **Mechanical polishing**
  - ▷ **Electro-polishing**
- **Removal of tenacious oxide layers (ex. Ti-, Al-rich)**
  - ▷ **Acidic solution**

During diffusion bonding

Variables (Temperature, external pressure, time)

With or without interlayer (ex. thin foil of Ni, Fe, Ni-Cr, Fe-Cr, etc)

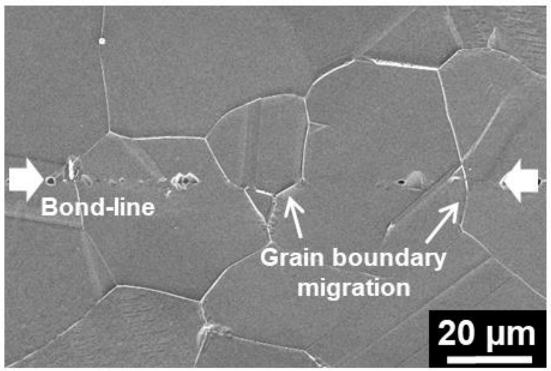
After diffusion bonding

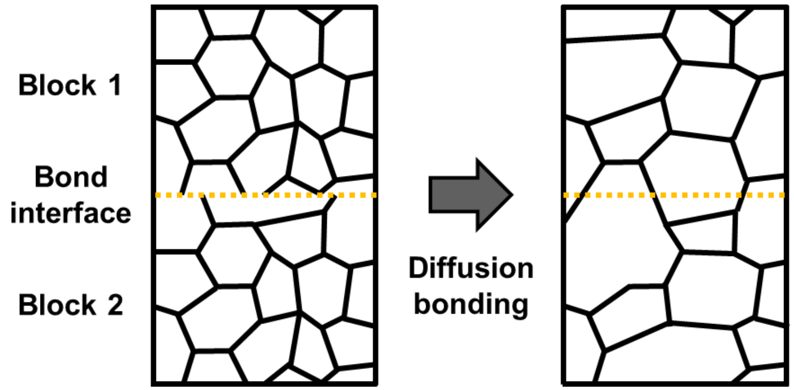
Post-bond heat treatment
- **Without external compressive pressure**

❑ Diffusion bonding of Fe- and Ni-base alloys

- Fe-base alloys : good bond efficiency with grain boundary migration across bond interface
- Ni-base alloys : poor bond efficiency → **Premature brittle bond-line fracture**

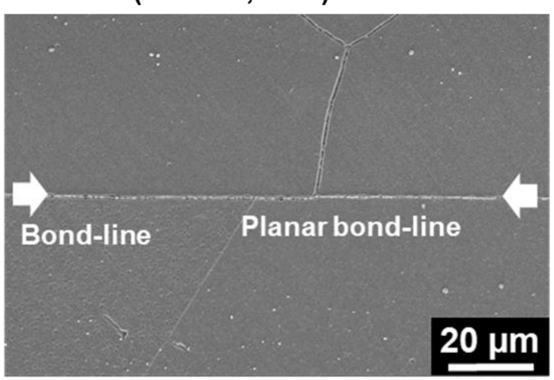
DB 316H (1050 °C, 8 MPa, 1 h)

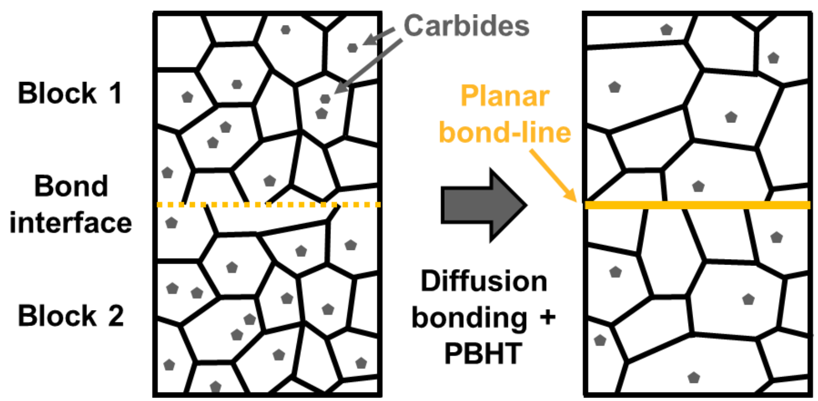




- Grain growth
- Grain boundary migration across bond-line
- Comparable tensile properties to as-received material
- Almost no change after S-CO_2 exposure or aging

DB 800HT (1150 °C, 10 MPa, 1 h)
+ PBHT (1100 °C, 20 h)





- Grain growth
- Planar bond-line due to carbide formation at bond-line
- Recovery of ductility after PBHT
- Loss of ductility after S-CO_2 exposure or aging

Research Objective

Corrosion and mechanical behavior of diffusion-bonded candidate alloys in high temperature S-CO_2 environment

Evaluation of diffusion-bonded joints for optimizing condition

→

Fabrication of Multi-Layered Diffusion-Bonded joints of candidate alloys

- Evaluation of microstructural and mechanical properties of MLDB joints compared to As-received

- Evaluation of corrosion resistance of MLDB joints after S-CO_2 exposure (600 ℃, 20 MPa, 500 h)

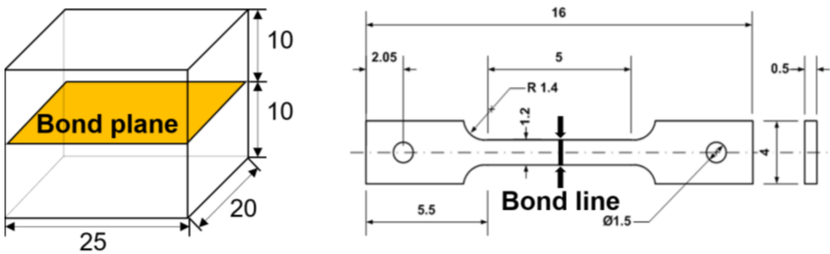
Experimental Procedure

❑ Candidate alloy

- Alloy 800H

❑ Diffusion bonding trials using small blocks

- Tensile properties (RT & HT) & microstructural analysis



▲ Schematics of blocks and mini-sized tensile specimens used for diffusion bonding trials

❑ Optimization of DB conditions for each alloy

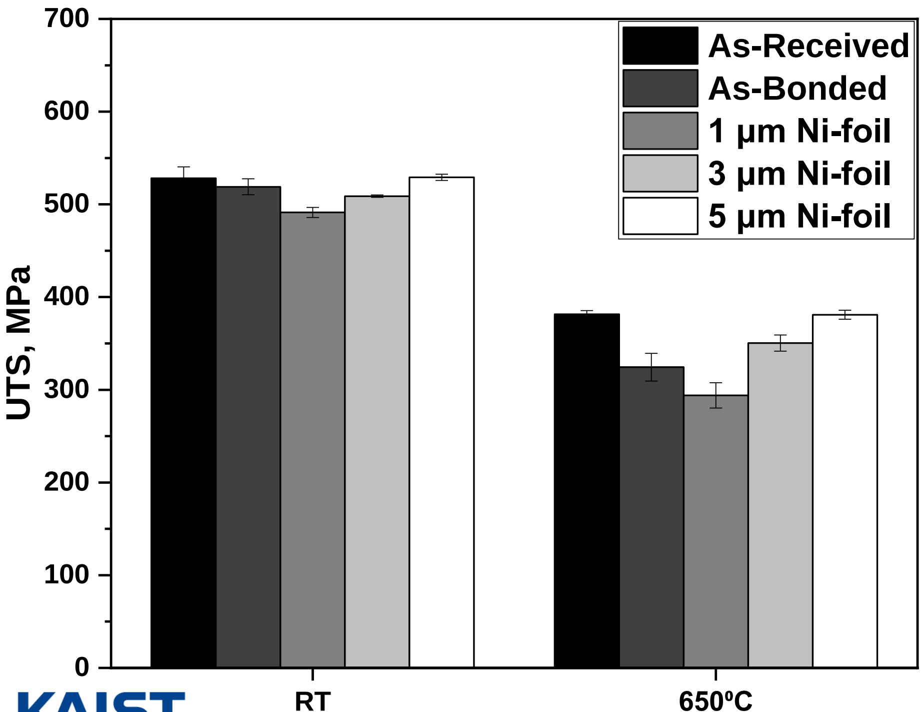
- Fabrication of multilayer diffusion bond joints using optimized conditions

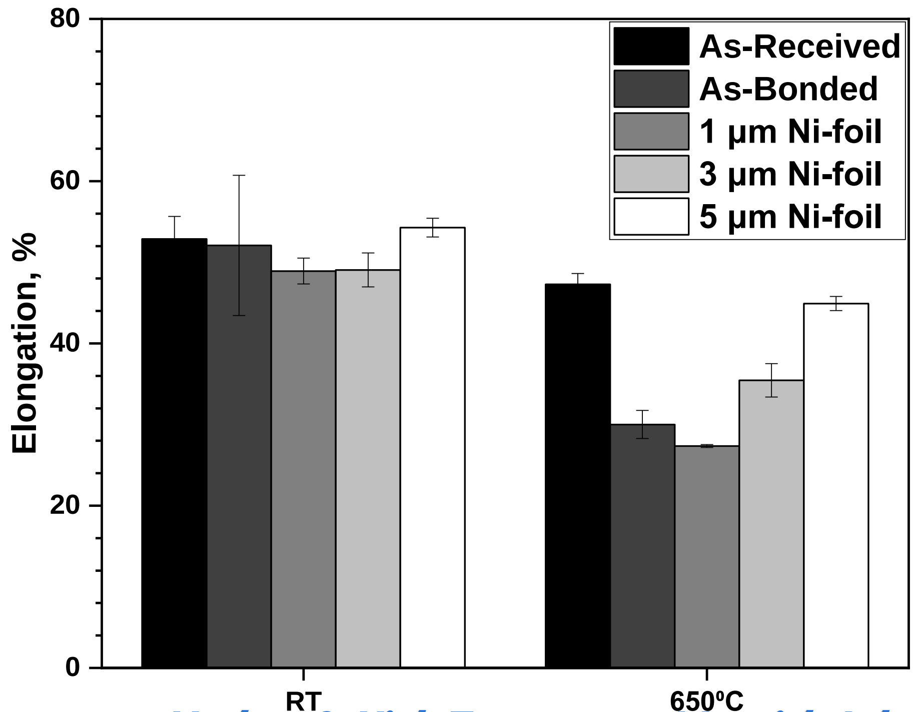
▪ DB conditions of candidate alloys

Alloy	Temperature (°C)	Pressure (MPa)	Duration time	Cooling	Surface condition	Interlayer	Post-bond heat-treatment (PBHT)
800H	1150	10	Hold temperature (10 min) + Pressure (60 min)	Furnace (vacuum) cooling	Mechanical Polishing (SiC grit #5000)	Ni-foil (0, 1, 3, 5 μm)	X
							1100 °C, 10 h (Air cooling) <HT-A Condition>
							1200 °C, 1 h (Air cooling) <HT-B Condition>

Results – Effect of Ni-Foil Interlayer on DB

- **Mini tensile test results of DB 800H : Thickness of Ni-foil interlayer**
  - DB conditions
    - 1150 ^{o}C, 10 MPa, 1 h, **Ni interlayers (0, 1, 3, 5 μm)**
  - No Ni interlayer & 1 μm Ni interlayer & 3 μm Ni interlayer
    - Brittle failure at bond-line & poor elongation compared to as-received condition
  - 5 μm Ni interlayer
    - **Similar tensile property compared to as-received condition**

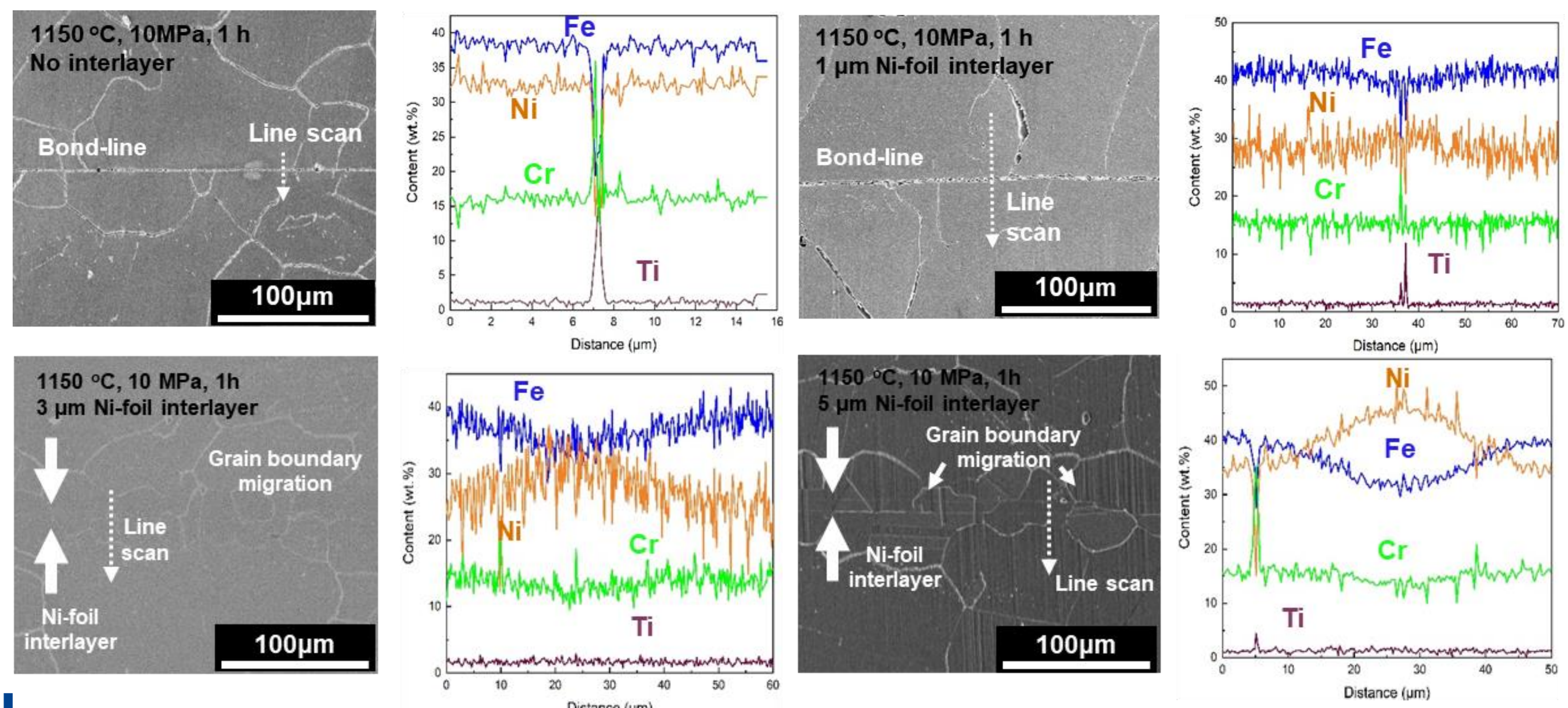




Results – Effect of Ni-Foil Interlayer on DB

❑ Microstructures of DB 800H : Thickness of Ni-foil interlayer

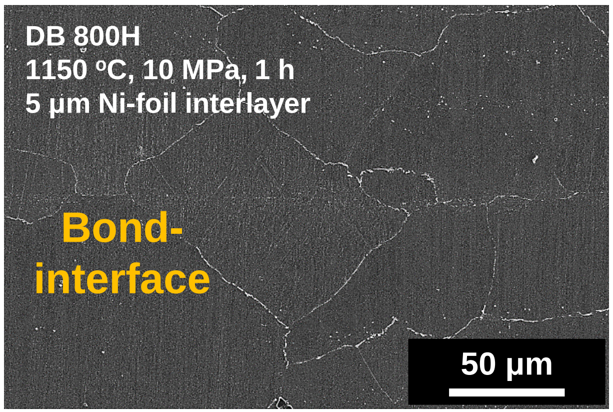
- No Ni interlayer & 1 μm Ni interlayer
  - Ti-rich carbide formation at bond interface → limit grain boundary migration
- 3 & 5 μm Ni interlayer
  - Suppress carbide formation at bond-line → grain boundary migration
  - Residual Ni at interlayer region → remove by applying PBHT

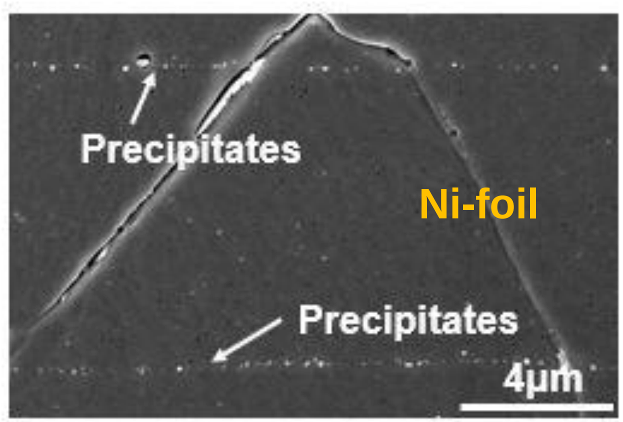


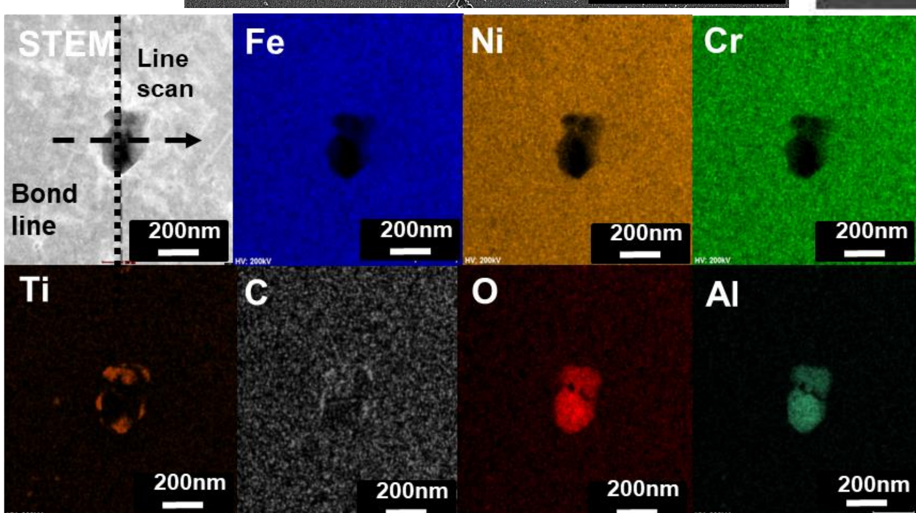
Results – Effect of Ni-Foil Interlayer on DB

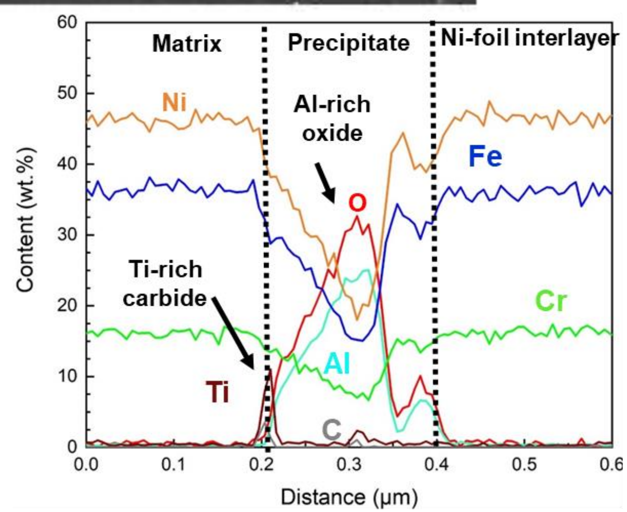
❑ TEM analysis of DB 800H (5 μm)

- Ni-foil interlayer (5 μm) significantly suppressed precipitate formation at DB line
- Still, small precipitates along Ni-foil / matrix interface (few hundreds of nm in size)
  - Al-rich oxides & Ti-rich carbides





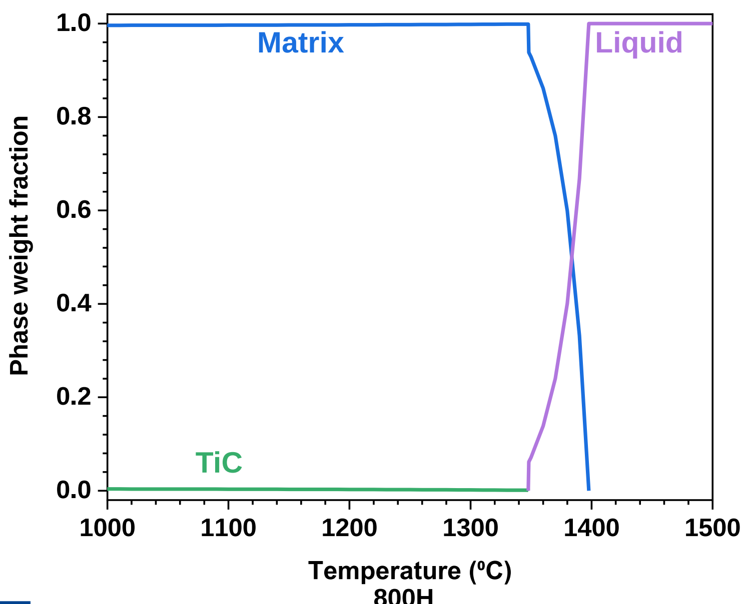


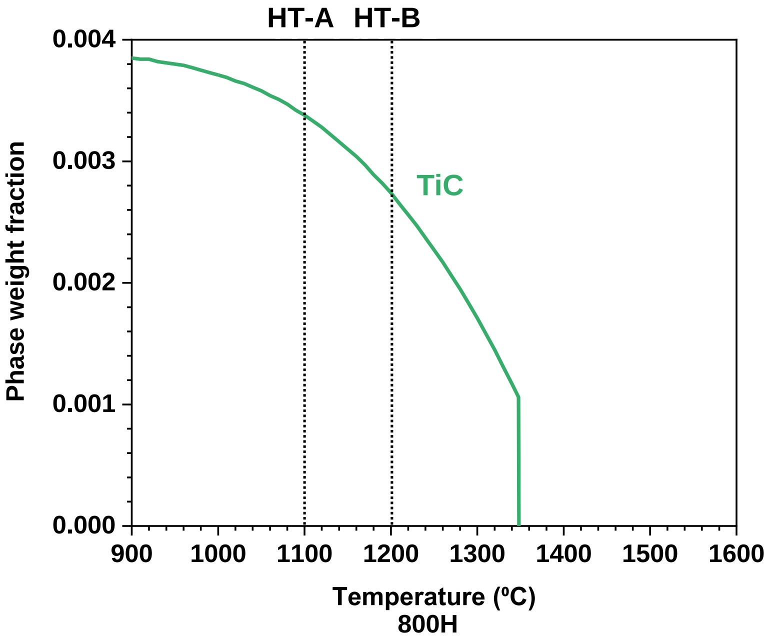


Results – Effect of PBHT on DB

❑ Selection of the PBHT conditions using Thermo-Calc software

- Dissolution temperature of precipitates using for chemical composition of alloy 800H
  - Phase stability calculation of the TiC precipitates in Alloy 800H
  - Diffusion of residual Ni and dissolution of Ti-rich precipitates at the bond-line
    - To induce the grain boundary migration
  - **Consider the grain growth and residual Ni on bond-line →HT-A condition**
  - **Consider the grain growth migration and fraction of TiC precipitates→HT-B condition**
    - **HT-A condition : 1100 ºC, 10 h**
    - **HT-B condition : 1200 ºC, 1 h**





Results – Effect of PBHT on DB

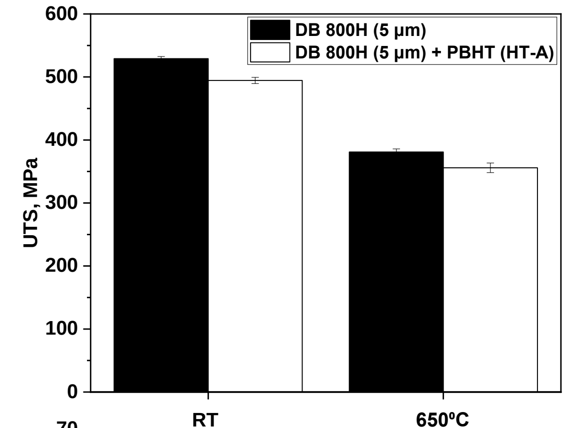
❑ Mini tensile test results of DB + PBHT (HT-A)

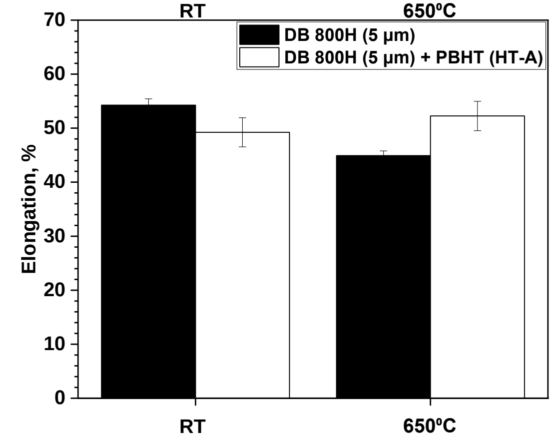
- Slightly decrease the tensile property
  - UTS (RT & 650 ℃), Elongation (RT)
- **Improve the elongation at 650 ℃ applied the PBHT**

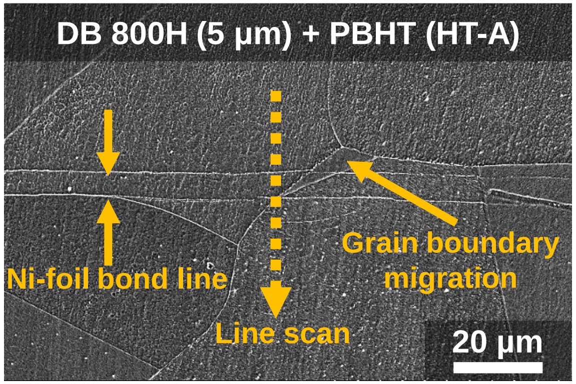
❑ Microstructures of DB + PBHT (HT-A)

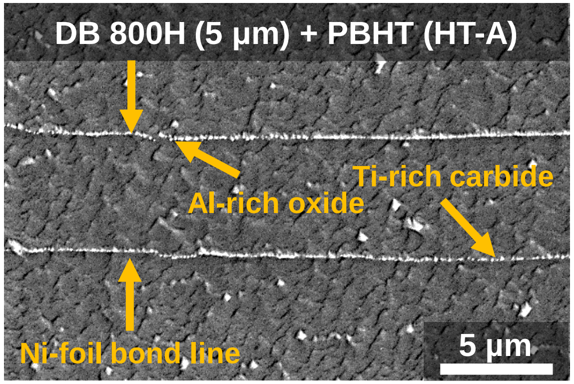
- Growth the grain size
- Diffusion of residual Ni
- Still, small precipitates along the interface

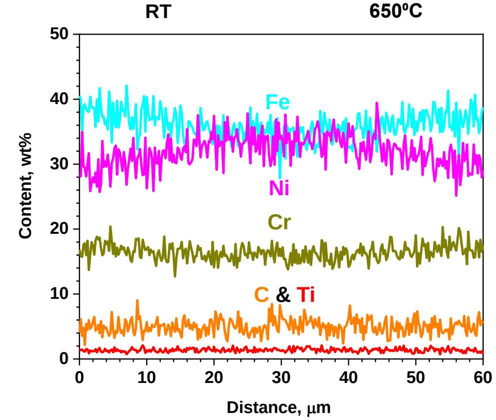
→ More grain boundary migration







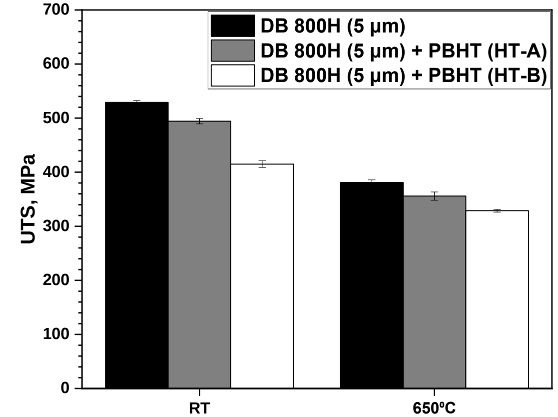


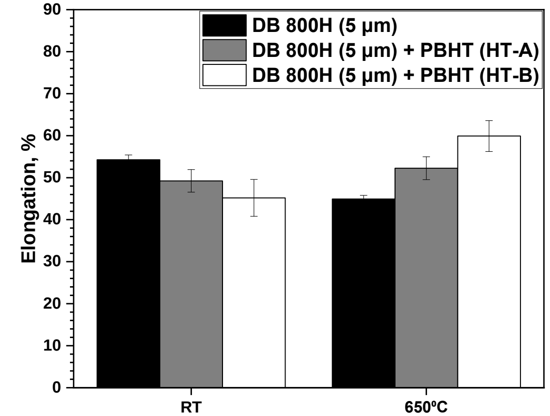


Results – Effect of PBHT on DB

❑ Mini tensile test results of DB + PBHT (HT-B)

- Slightly decrease the tensile property
  - UTS (RT & 650 ℃), Elongation (RT)
- **Improve the elongation at 650 ℃ applied the PBHT**

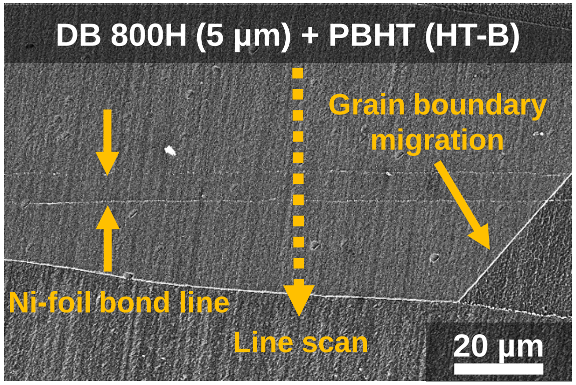


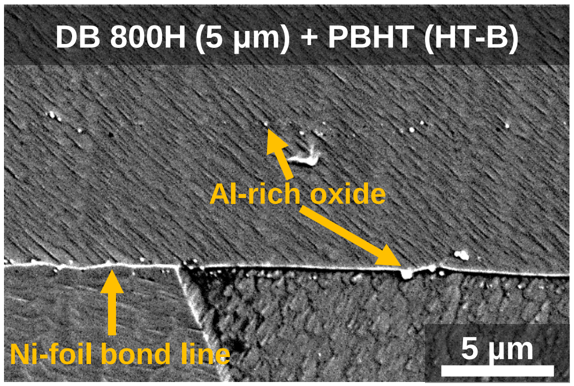


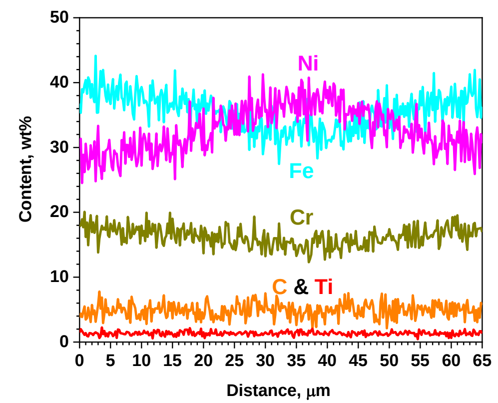
❑ Microstructures of DB + PBHT (HT-B)

- Growth the grain size
- Partial diffusion of residual Ni
- Disappear the Ni-foil bond line
  - **Dissolution of Ti-rich precipitates on bond line**

→ Smooth the grain boundary migration







Summary

- **Corrosion and mechanical behavior of diffusion-bonded candidate alloys in high temperature S-CO_2 environment**
  - Characterization of diffusion-bonded joints
    - Fe-base alloys : good bond efficiency with grain boundary migration across bond interface
    - Ni-base alloys : poor bond efficiency → **Premature brittle bond-line fracture**
  - Effect of thickness of Ni-foil interlayer (0,1,3,5 μm)
    - No Ni interlayer & 1 μm Ni interlayer
      - Brittle failure at bond-line & poor elongation compared to as-received condition
    - 3 & 5 μm Ni interlayer
      - Suppress carbide formation at bond-line → grain boundary migration across bond interface
  - Effect of Post-Bond Heat Treatment on DB 800H
    - **HT-A condition** → grain boundary migration across bond-line & improved the elongation
      - **Focus on the diffusion of residual Ni**
    - **HT-B condition** → grain boundary migration across bond-line & improved the elongation
      - **Focus on the dissolution of Ti-rich precipitates on bond-line**

Precipitates on the bond-line after diffusion bonding are crucial for long-term DB joint integrity of Alloy 800H

Further work

❑ Further work

- Fabrication of MLDB with optimized conditions
  - 1150 ^{o}C, 9 MPa, 1 h + PBHT (1200 ^{o}C, 1 h)
- Air creep tests
  - Focus on the creep-rupture time compared to AR and Not PBHT specimens
- Corrosion and SCC tests in S-CO_2 (600 ^{o}C, 20 MPa, 500 h)
  - Focus on the corrosion and SCC behavior of DB joints compared to AR
- Development & optimization of model alloys designed for DB
  - Focus on the Al- and Ti- content of chemical composition

Energy for Earth !!

Thank you!

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Appendix I. Chemical compositions

❑ Chemical compositions of candidate materials

wt.%	Fe	Cr	Ni	C	Ti	Mo	Mn	Al	Si	Other
800H	Bal.	20.12	31.85	.07	.51	-	.87	.49	.17	
800HT	Bal.	21.0	33.6	.06	.55	0.2	0.9	.48	0.4	.003 B .05 Co 0.1Cu
316L	Bal.	16.74	10.09	.016	-	2.04	1.28	.03	.34	N: .07 Co: .19
316H	Bal.	17.3	10.7	.05	-	2.1	0.6	-	0.6	0.2 Cu