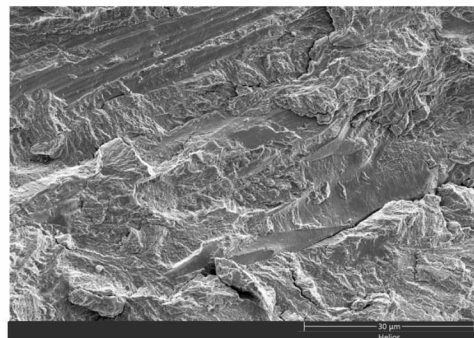
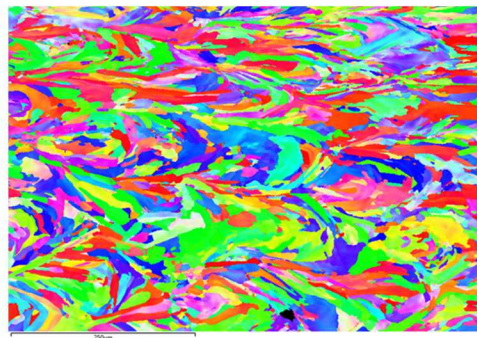




# Hydrogen-assisted fracture of additively manufactured austenitic stainless steels

Chris San Marchi, Josh Sugar, Dorian Balch  
Sandia National Laboratories, Livermore CA

3<sup>rd</sup> International Conference on Metals and Hydrogen  
29-31 May 2018  
Ghent, Belgium





# Motivation

- **Additive manufactured austenitic stainless steels show interesting strength characteristics**
  - Questions remain about reproducibility of materials and microstructures
  - Defects appear to be an intrinsic characteristic
- **New design paradigm with additive is particularly attractive for demanding applications in extreme environments**
  - For example, applications that require hydrogen compatibility
- **Many additive processes are intrinsically solidification based**
  - Therefore, knowledge of the performance of weld microstructures may be transferrable





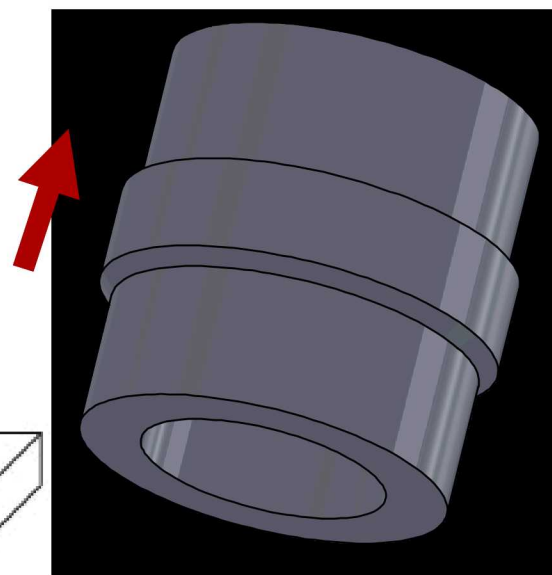
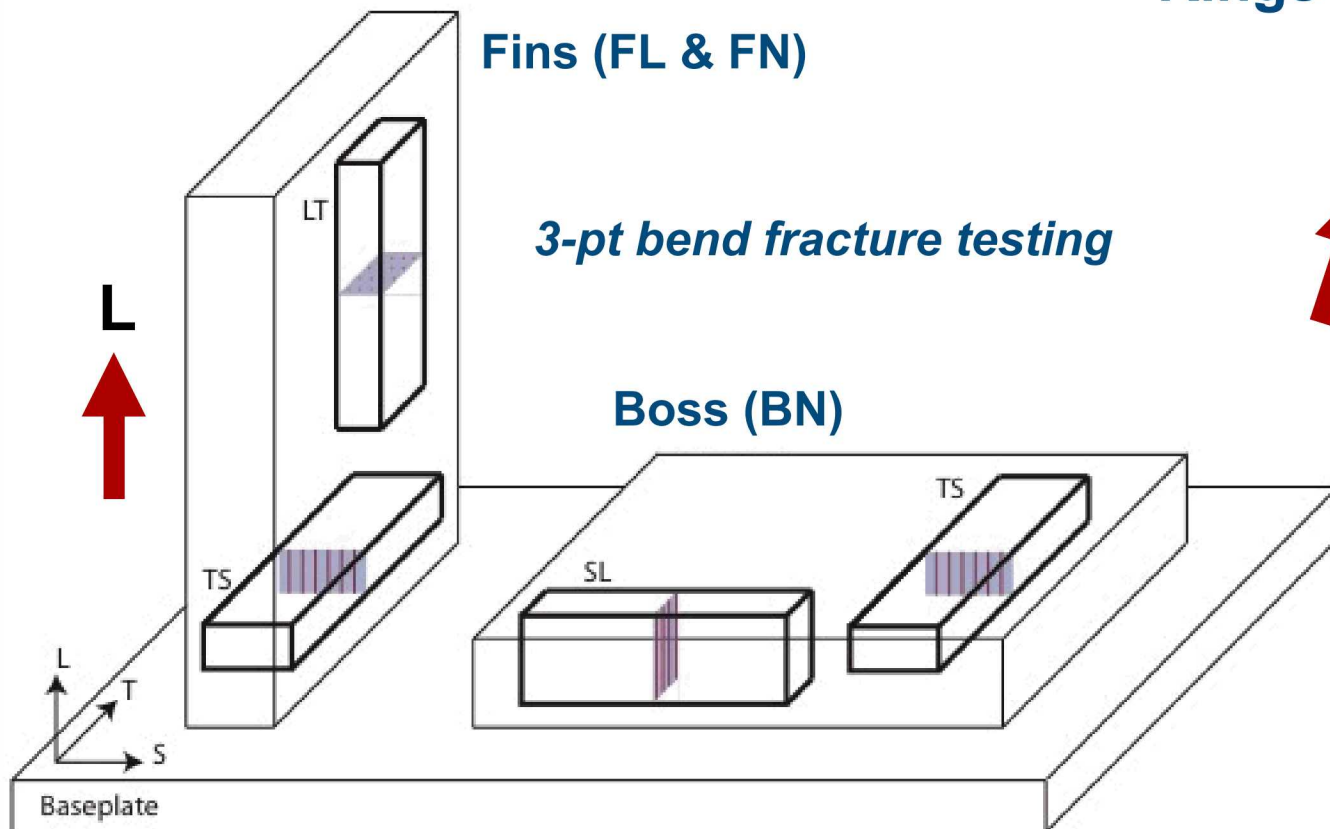
# Materials: additively manufactured (AM) 304L

## Directed-energy deposition (DED)

- Fins (FL)

## Powder bed fusion (PBF)

- Fin (FN)
- Boss (BN)
- Rings (RL and RN)



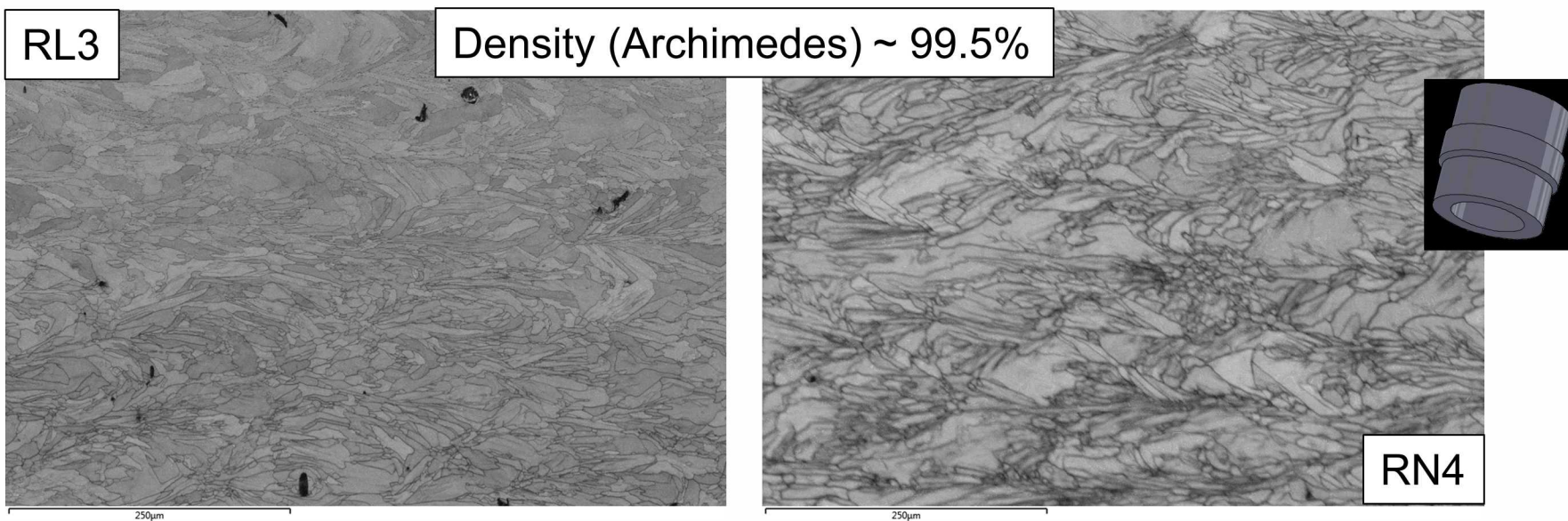
Rings (RL & RN)

*Tensile testing*



# Composition of AM 304L is typical of wrought materials, except oxygen and nitrogen

	Fe	Cr	Ni	Mn	Si	C	N	O	S	P
powder	bal	18.35	9.69	1.37	0.57	0.012	0.096	0.042	0.008	0.012
RL1	bal	18.55	9.58	1.37	0.59	0.013	0.050	0.042	0.004	0.012
RN1	bal	18.35	9.92	1.41	0.54	0.010	0.047	0.043	0.004	0.013

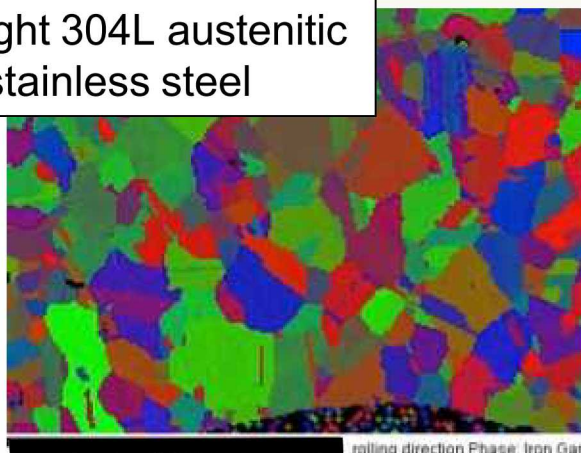






# AM microstructure is similar to weld microstructure, but finer scale

Wrought 304L austenitic  
stainless steel



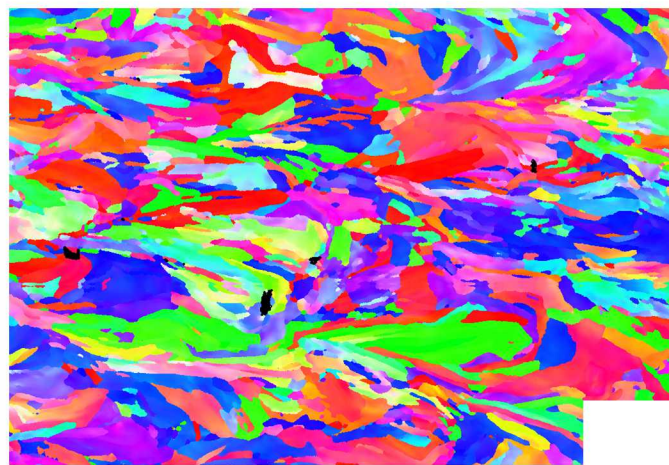
100 μm

Gas tungsten arc weld 304L/308L



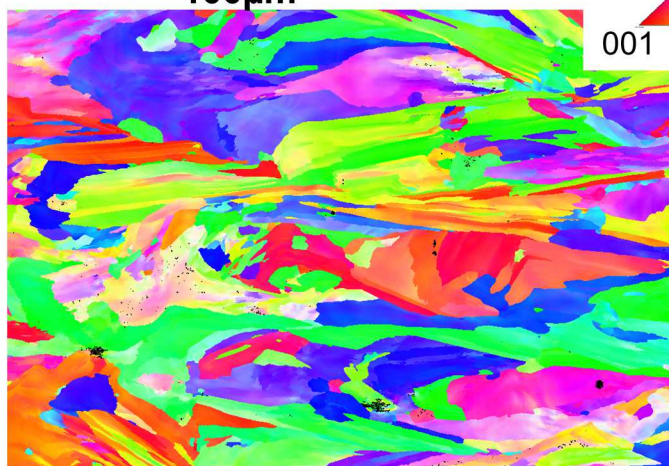
2.5 mm

Inverse pole figures

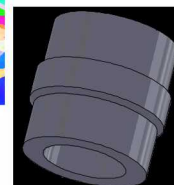
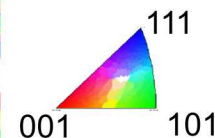


RL3

100 μm

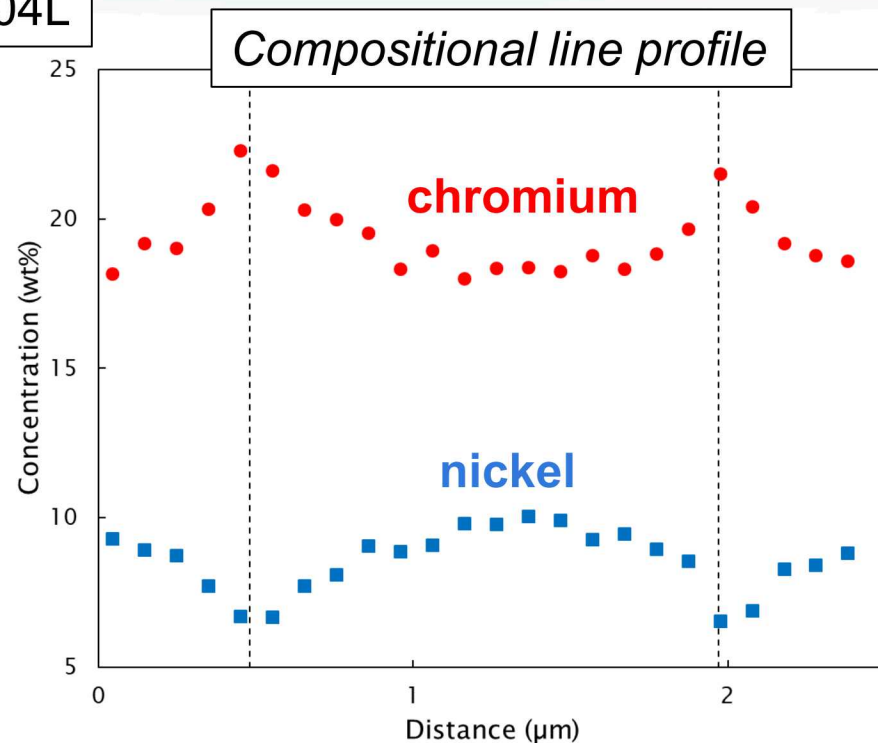
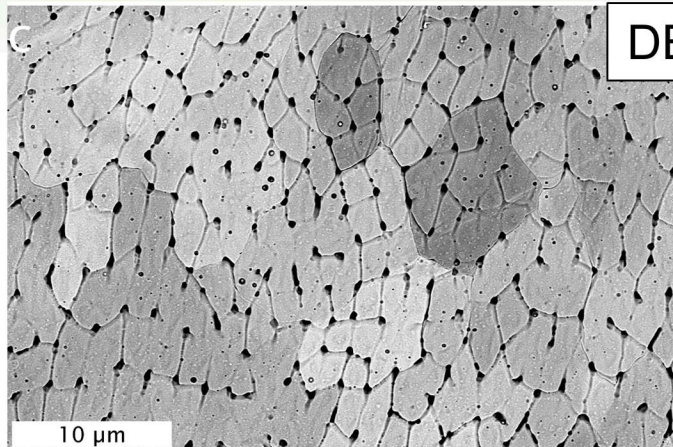


RN4





# Compositional microsegregation in AM 304L can be significant



Courtesy of Thale Smith, UC Davis/Sandia

- Solidification microstructure leads to compositional segregation on fine length scale
- Ferrite content is typically low
  - DED < 2%
  - PBF < 0.5%

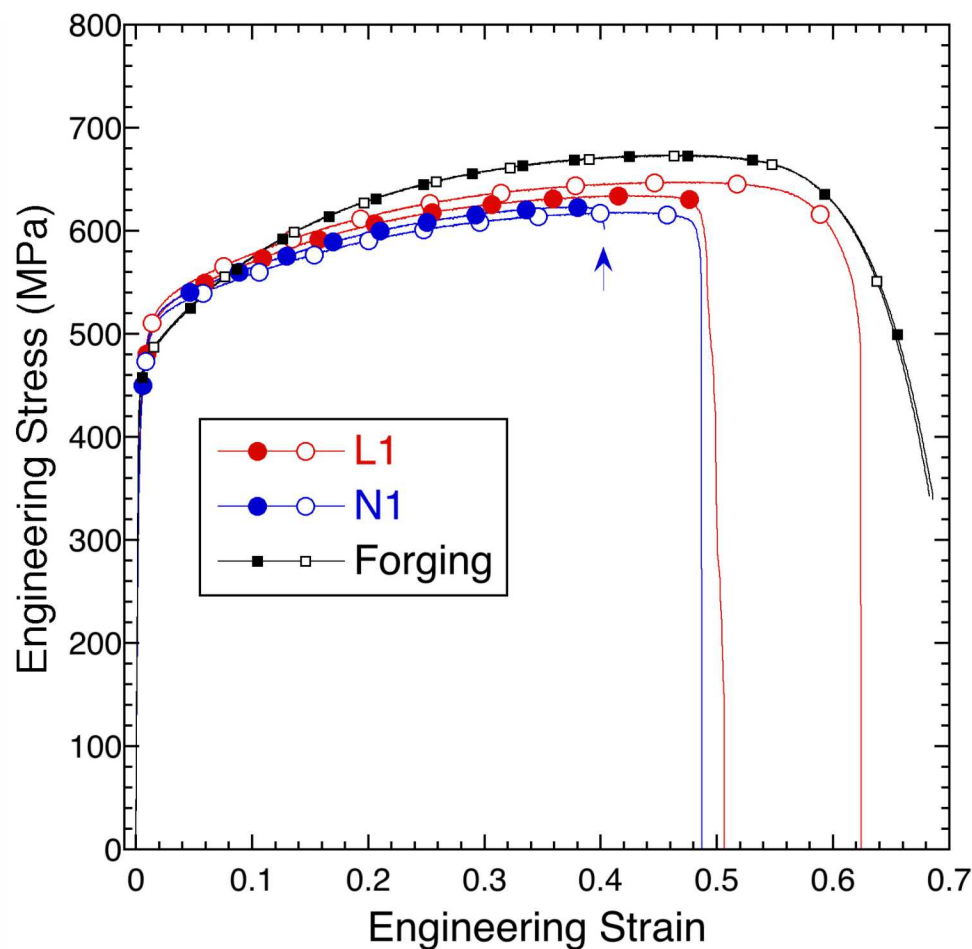
**Segregation of nickel (in particular) can have important implications on hydrogen-assisted fracture**

# Tensile strength of AM materials reflects strain-hardened condition

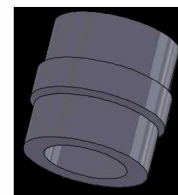
Build geometry	Designation	YS (MPa)	TS (MPa)	EI (%)	RA (%)
DED Fin	FL1	441	686	Estimated from hardness measurements	
DED Fin	FL2	372	650		
PBF Fin	FN	348	638		
PBF Boss	BN	292	612		
PBF Ring	RL1	414	641	57	49
PBF Ring	RN1	413	621	45	39
Forging	55C	452	674	68	81

- ASTM E8 subsized cylindrical tensile specimens: 4 mm dia, 19 mm gauge
- Nominal strain rate  $10^{-3} \text{ s}^{-1}$  (constant crosshead)

# Strength properties are consistent, ductility is variable



- Elongation to failure of PBF 304L is lower than expected for wrought 304L
- Elongation varies between 40-60% for RL and RN

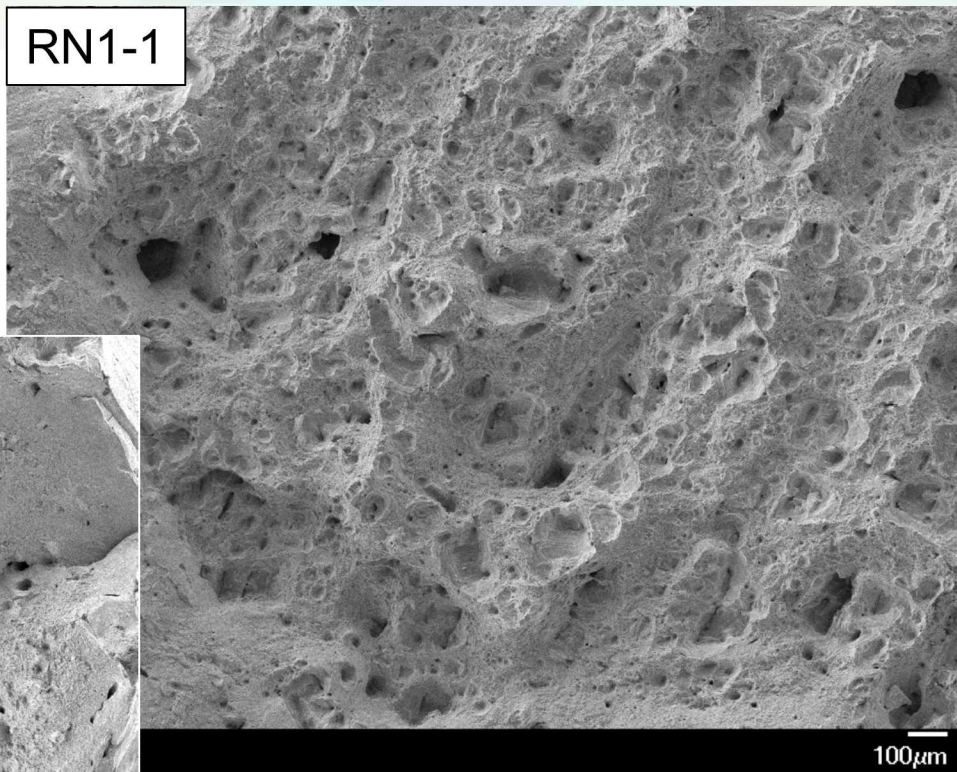
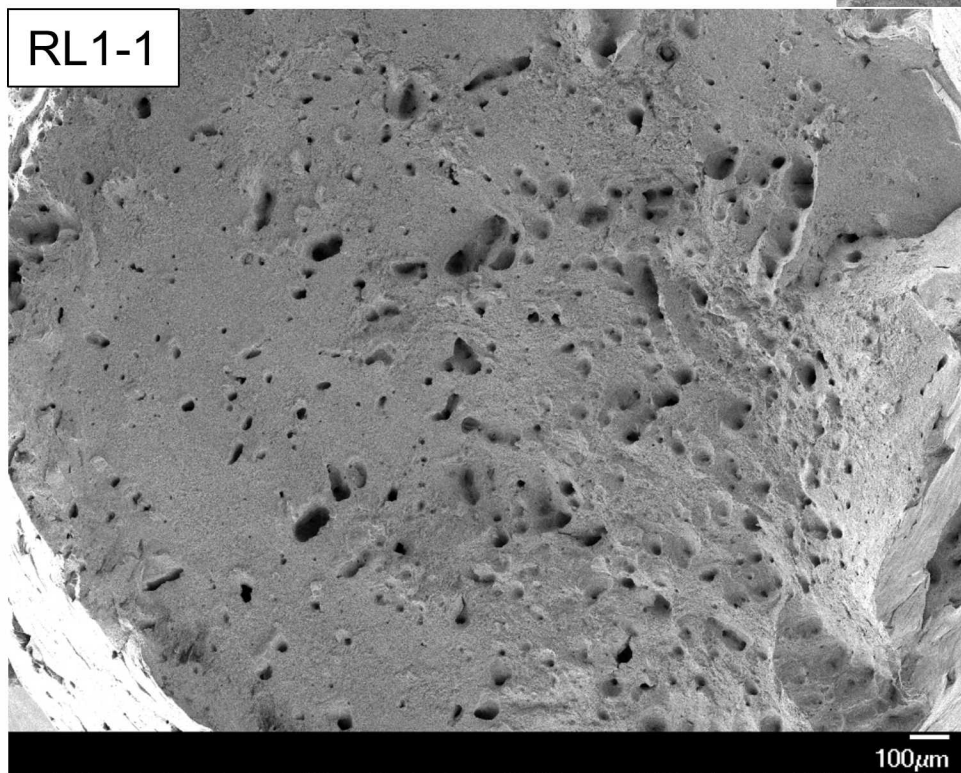




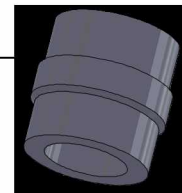


# Fractography reveals defects: probable source of relatively low reduction of area

**RL1 material appears to have moderate density of distributed defects**

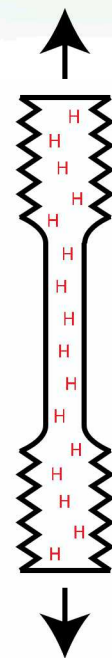
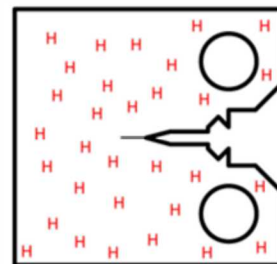


**RN1 material appears to have higher density of interacting defects**



# Testing in the H-precharged condition

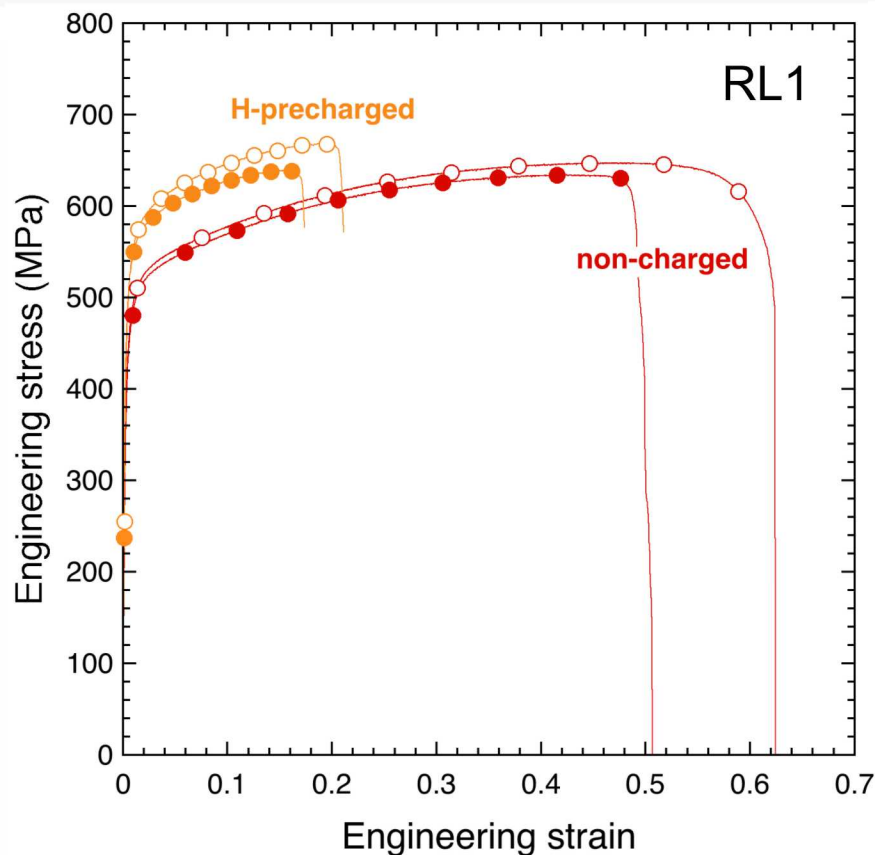
- Thermal H-precharging
  - Exposure to gaseous H<sub>2</sub> until saturated with hydrogen (~60 days)
    - Pressure: 138 MPa
    - Temperature: 300°C
  - Hydrogen content ~140 ppm (wt)
- Testing in air after precharging with hydrogen



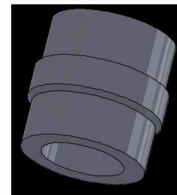
- Mechanical testing in H-precharged condition is similar to *in situ* testing in high-pressure gaseous hydrogen for tension, fatigue and fracture
  - *Must consider the H-solute hardening: strength increase of 10-20%*



# Ductility of PBF 304L is severely degraded when H-precharged



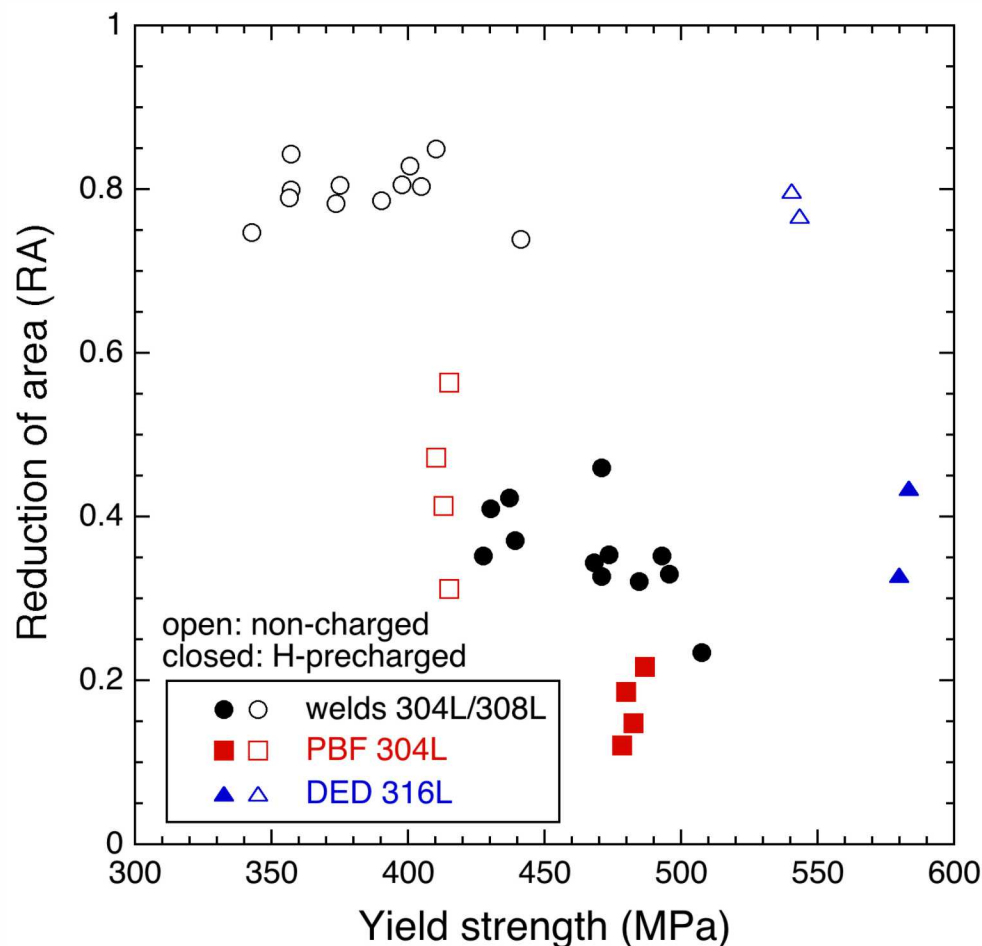
- Ductility of PBF 304L is significantly lower than forged 304L in the absence of hydrogen
  - RA = 40-50% (AM) vs RA = 80% (forged)
- Hydrogen reduces ductility in PBF 304L more than in forged 304L
  - RA = 13-20% (AM) vs RA = 50+% (forged)



**Ductility is lower than expected for H-precharged 304L**

# Tensile ductility is strongly affected by defects in AM austenitic stainless steels

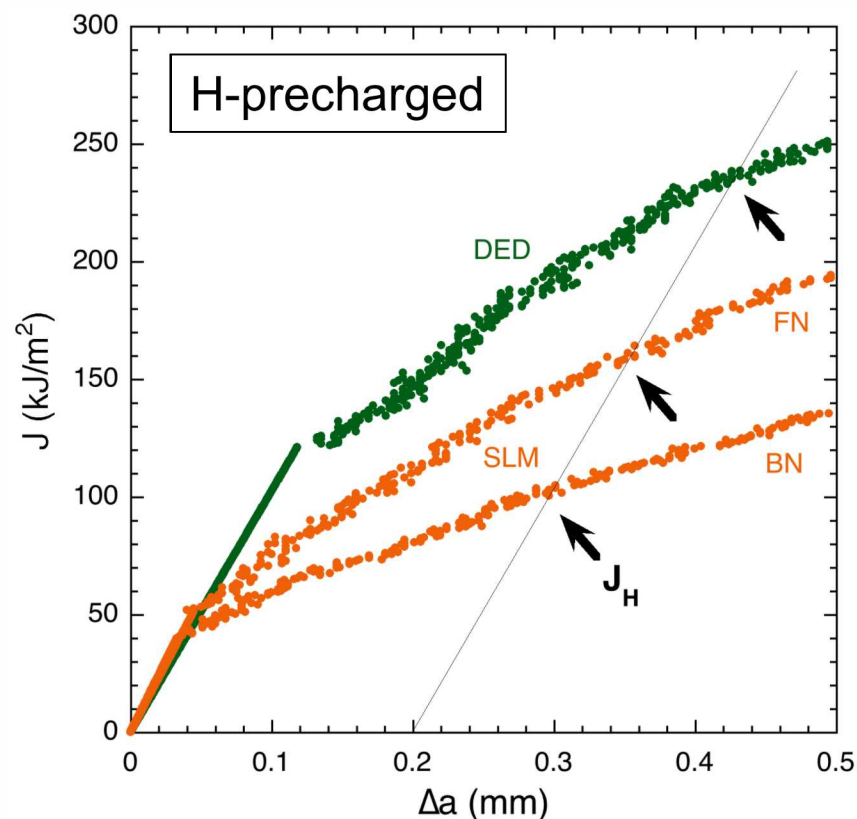
- Ductility of welds and high-quality AM materials is similar to wrought
  - RA ~ 80%
- Defects significantly reduce tensile ductility of AM materials
- Tensile ductility of AM in the presence of hydrogen reflects initial ductility
  - Relative reduction of ductility due to hydrogen appears to be independent of defects



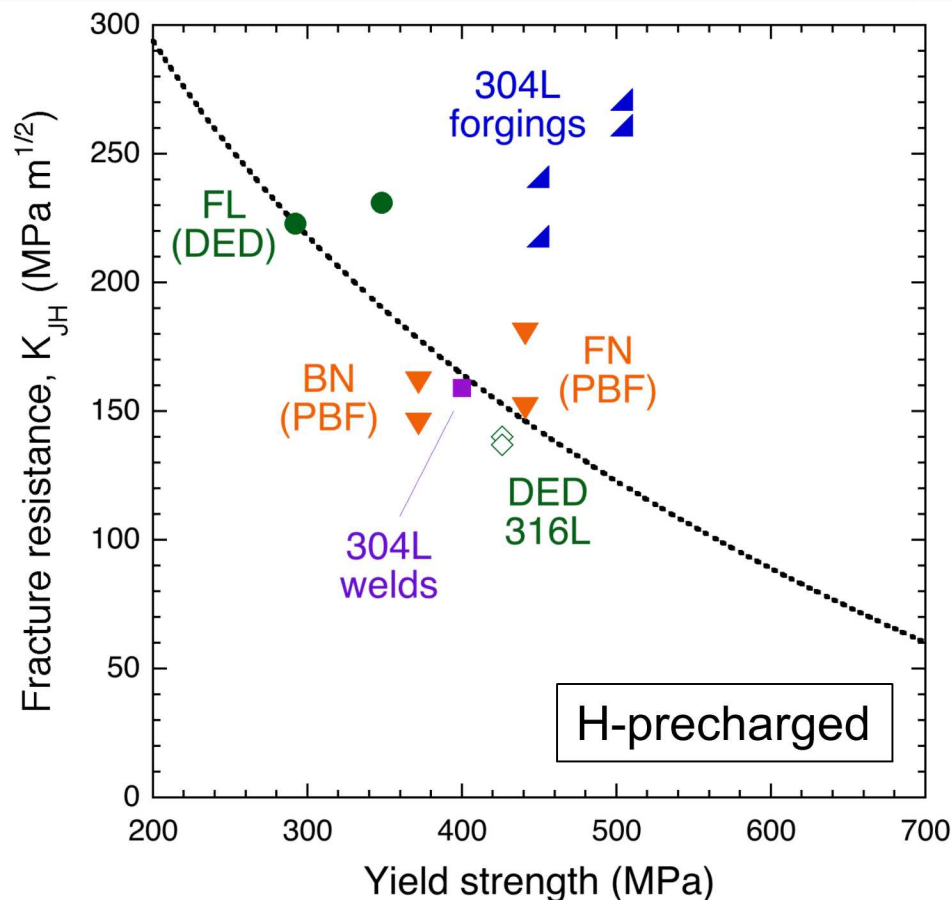


# H-assisted fracture in PBF 304L austenitic stainless steel is similar to welded material

- Elastic-plastic fracture resistance (JR curve)
- 3-pt bend specimens
  - $W = 12.7$  mm
  - $B = 6.3$  mm
  - $B_N = 4.9$  mm
- Monotonic loading using direct current potential difference (DCPD) to monitor crack extension ( $\Delta a$ )
- Constant displacement rate corresponding to  $< 3 \text{ MPa m}^{1/2}$  per minute



# H-assisted fracture in PBF 304L austenitic stainless steel is similar to welded material



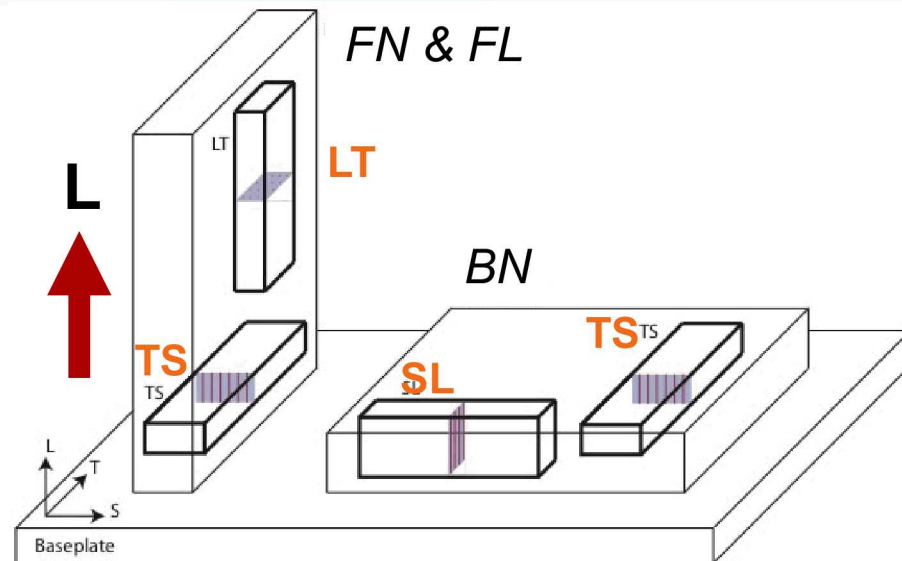
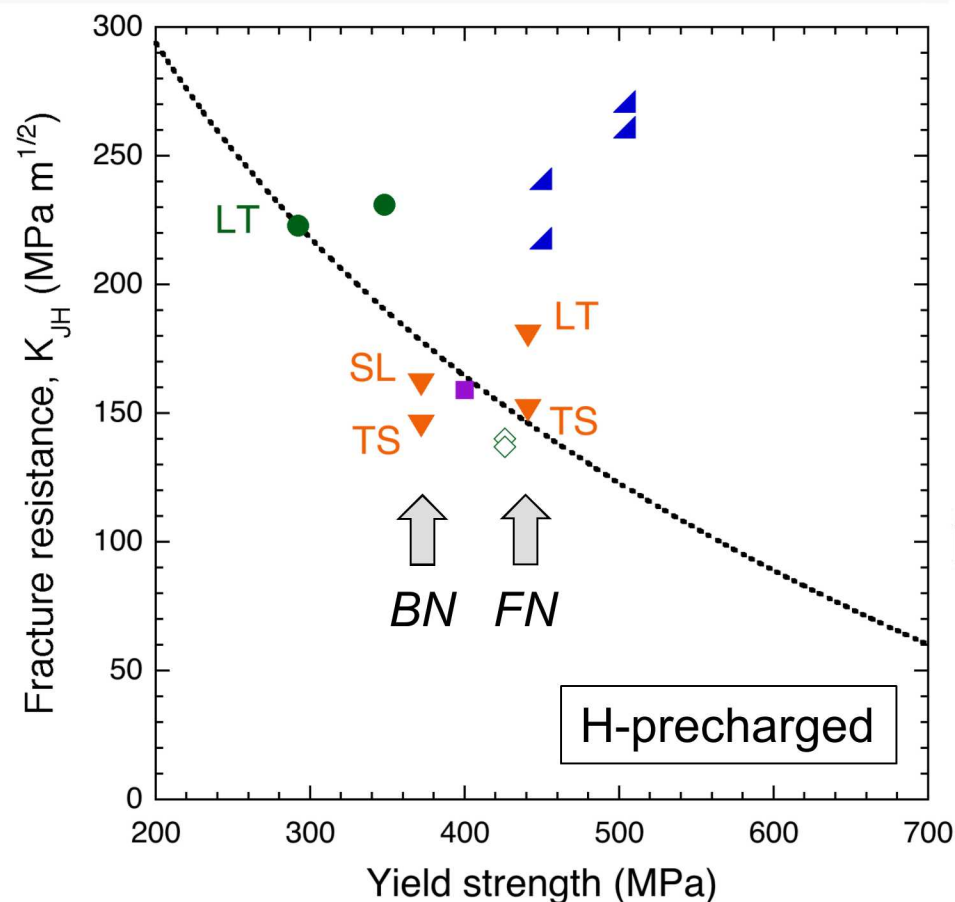
- Fracture toughness of AM stainless steels can be very high in air (not shown but >300 MPa m<sup>1/2</sup>)
- When H-precharged, fracture resistance of AM materials is less than forged material
- AM materials appear consistent with:
  - Welds
  - Expected trend with strength

304L forgings: Jackson, Metall Mater Trans 47A

Welds: Jackson, Corros Sci 60



# Orientation has a relatively modest effect on H-assisted fracture of PBF 304L

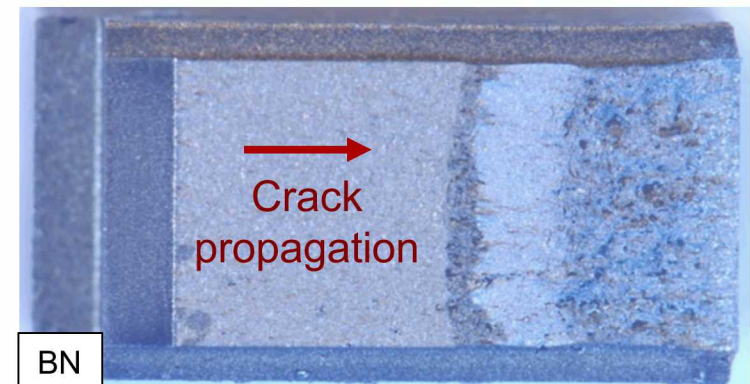
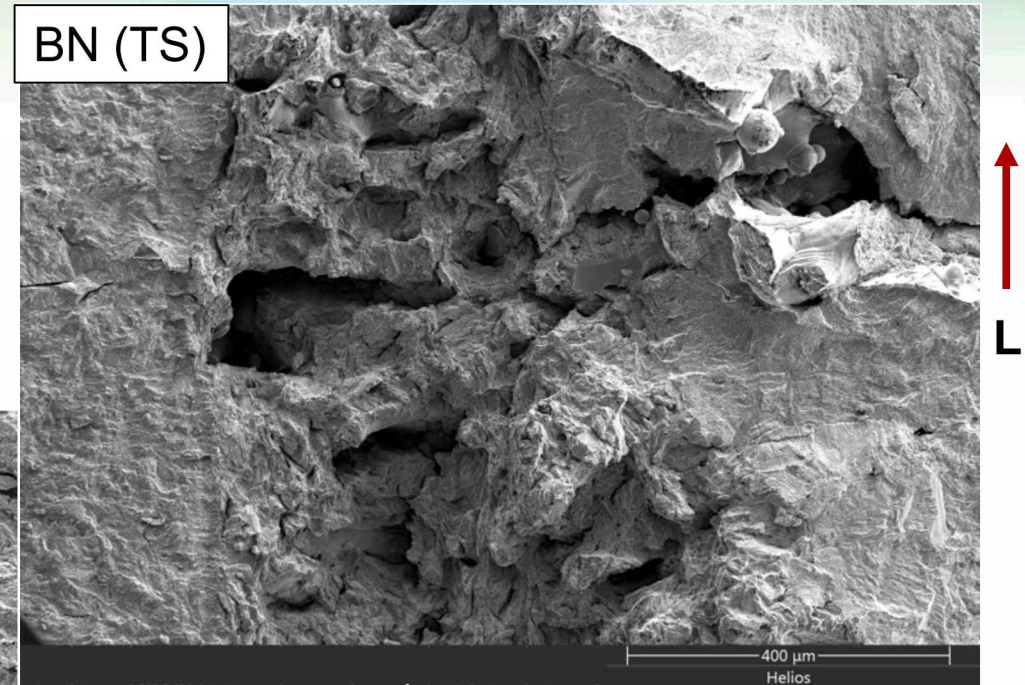
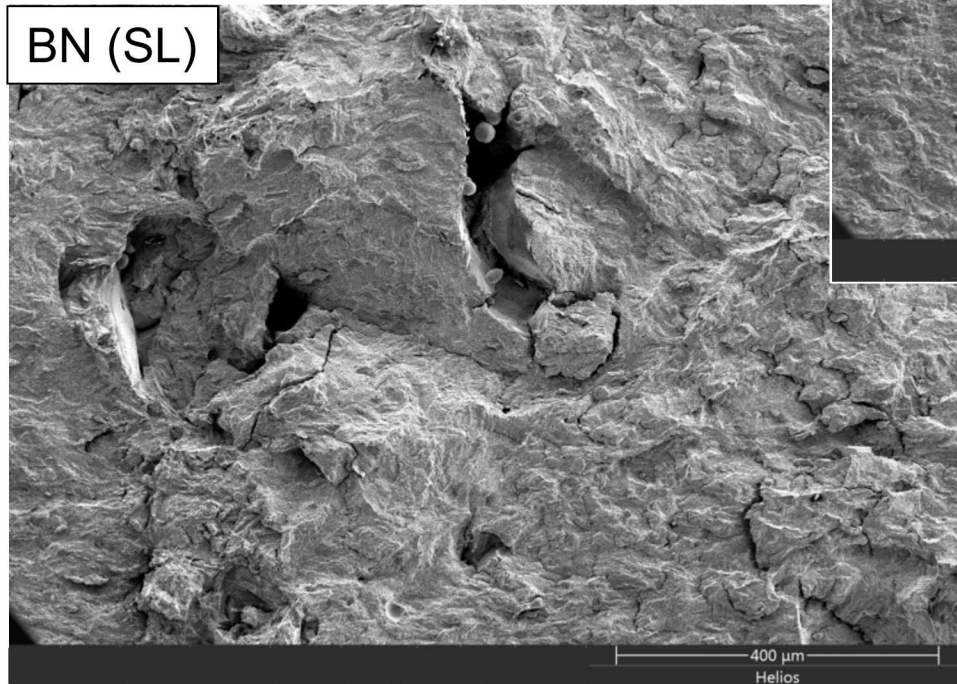


- Fracture resistance appears to be lowest in TS orientation
- Fracture resistance is greatest when crack plane normal is build direction (L)

# Fractography illuminates defect structure

- Macroscopic defects are associated with interlayers of build (lack of fusion)

→ L

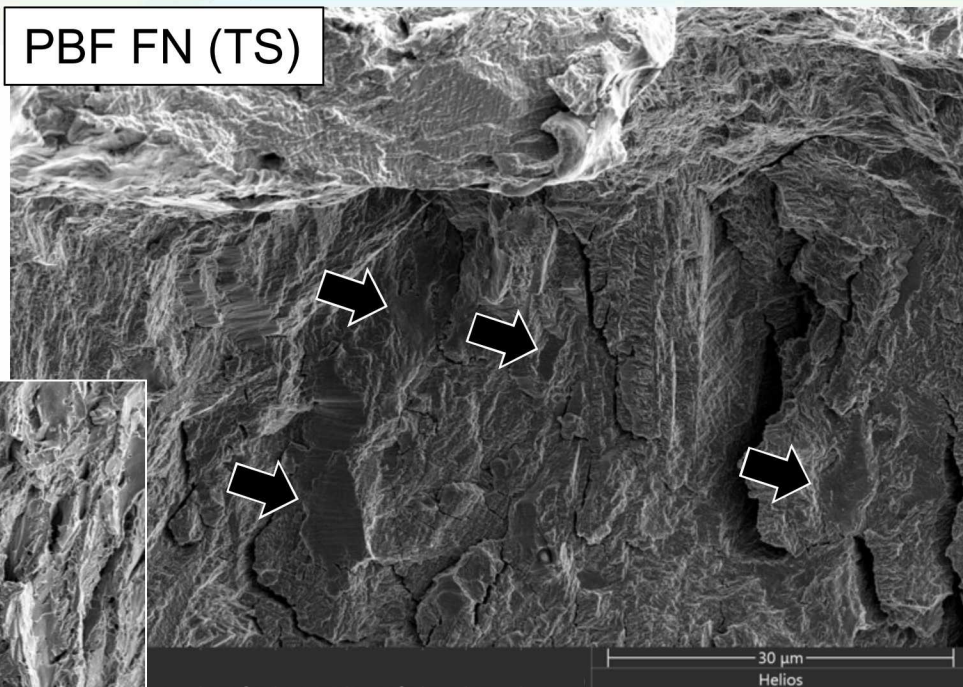




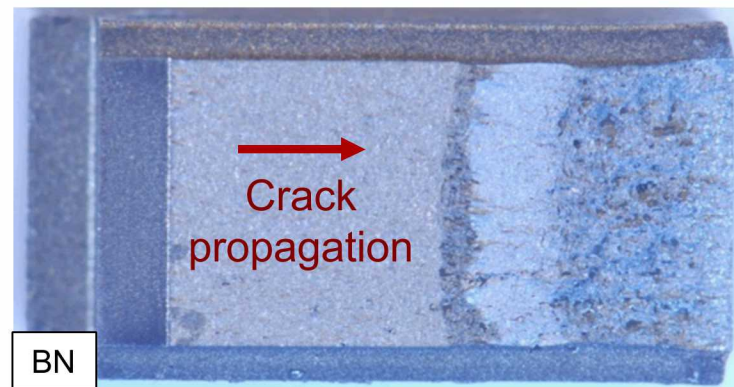
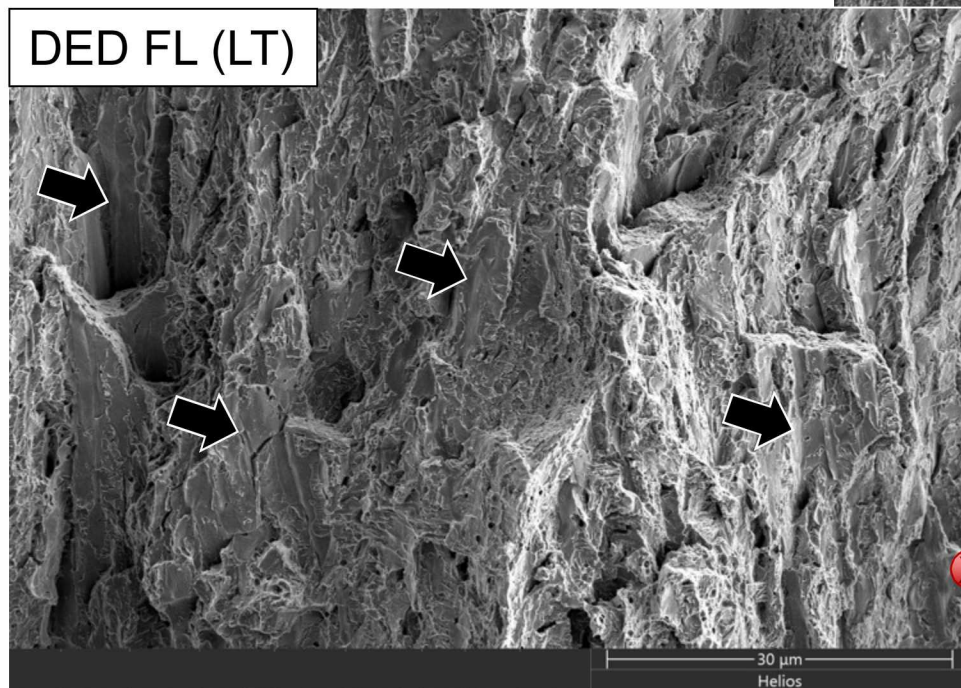
# Fine fracture features consistent with H-assisted fracture and microstructure

- Flat facets may represent solidification features →
- Lack of fusion defects do not play an obvious role

PBF FN (TS)

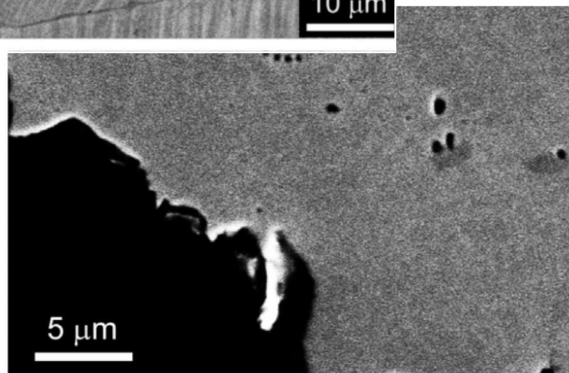
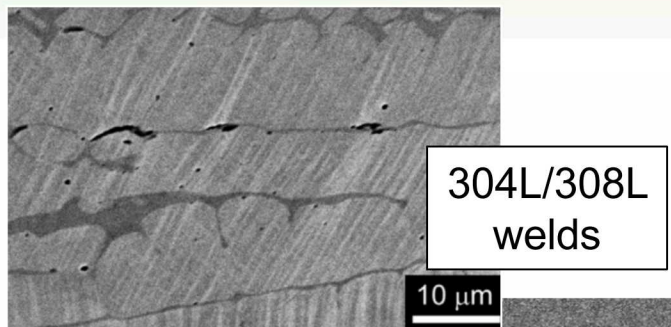


DED FL (LT)



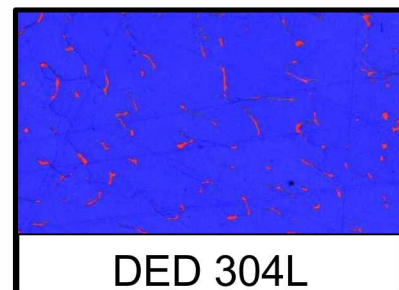


# Mechanisms of fracture may be similar to observations for welds



H-precharged 304L/308L welds show:

- Fracture of ferrite
- Fracture at  $\gamma/\delta$  boundaries
- Void nucleation at ferrite boundaries

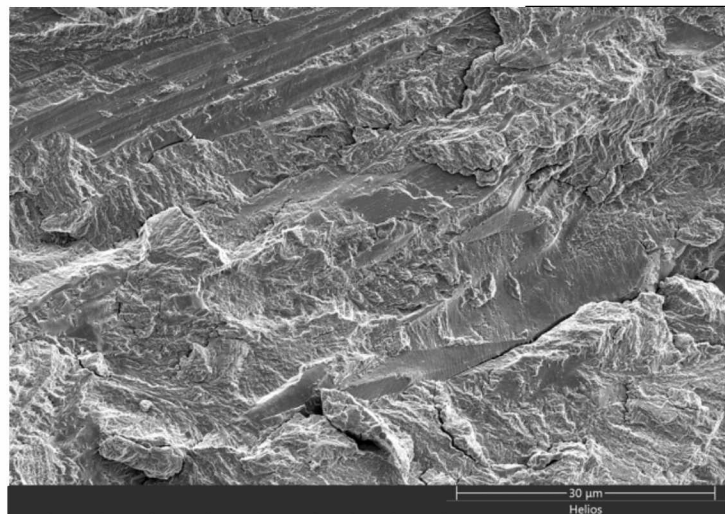


PBF material shows:

- Elongated flat fracture features that may be related to underlying solidification structure

from: Jackson, Corros Sci 60

Like in welds, hydrogen-assisted fracture in AM austenitic stainless may be related to *ferrite* and *compositional microsegregation*





# Summary

- **PBF 304L features strength consistent with strain-hardened (forged) wrought 304L**
  - **Strength properties can be reproducible**
  - **Ductility varies significantly in PBF materials**
- **PBF 304L is more sensitive to H-assisted fracture than wrought materials**
  - **Significant degradation of elongation and RA**
  - **Fracture resistance is lower than wrought material when H-precharged, but consistent with welds**
- **Mechanisms of hydrogen interactions appear qualitatively similar to welded microstructures**

