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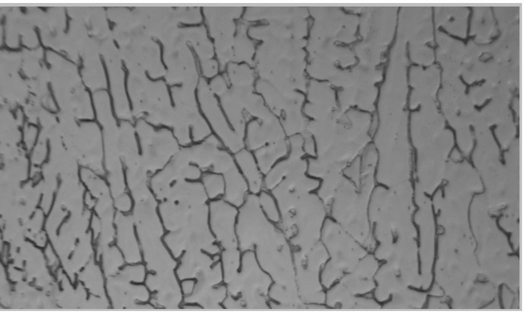

Temperature Effects on Fracture Thresholds of Hydrogen Precharged Stainless Steel Welds

Joe Ronevich, Chris San Marchi, Dorian Balch

Sandia National Laboratories, Livermore, CA USA

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Purpose: Evaluate low temperature fracture behavior of stainless steel welds saturated with hydrogen

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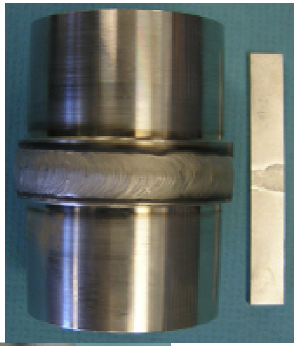
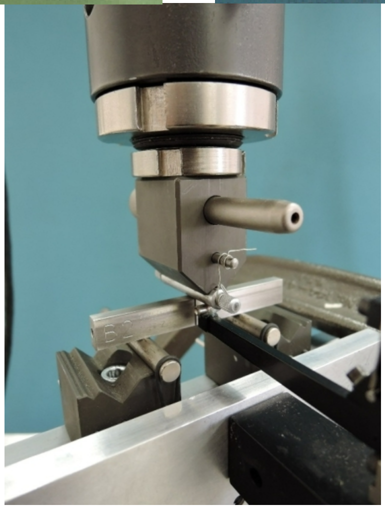
- Austenitic stainless steels are generally resistant to H_2 embrittlement
  - Desirable for H_2 containment components
  - However, welds can be more vulnerable
- *Low temperature* fracture behavior is not well characterized, particularly of welds
  - Many components in hydrogen refueling infrastructure are exposed to sub-ambient temperatures
- Tensile data of 300 series stainless shows degradation in elongation at sub-ambient temperatures, but fracture data not fully characterized

Reliable measurements of H_2 effects on welds needed to ensure integrity of pressure vessels and improve design margins

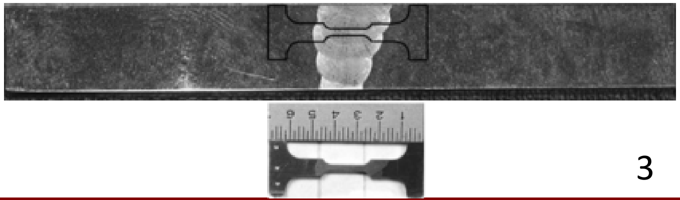
Approach: Fracture tests of H_2 precharged stainless steel welds at sub-ambient temperatures

- Extracted 3-point bend bars from forged gas tungsten arc welded (GTAW) rings
  - 304L
  - 316L
  - XM-11 (21Cr-6Ni-9Mn)
    - All welded with 308L filler metal
- Machined notch centered in weld
- Thermal precharge with H_2
  - 300°C for 16+ days at 138 MPa
- Elastic-Plastic Fracture (J-R curves) tests of welds at 223 K and 293 K
  - Liquid Nitrogen chilled environmental chamber





- Extract mini-tensile specimens from weld region for testing at 223 K and 293 K

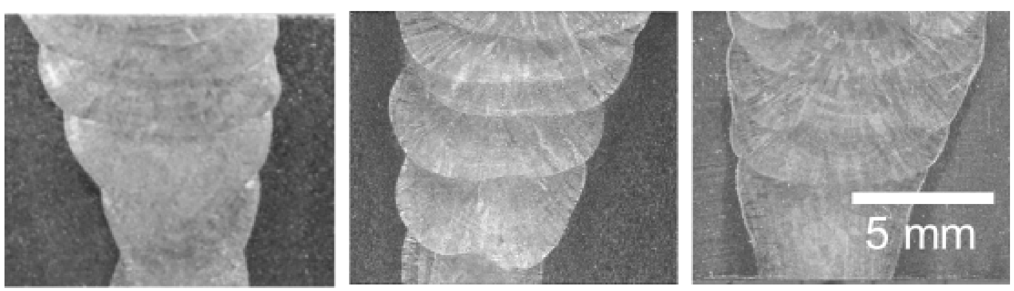


Materials: Forged Austenitic Stainless Steel Gas Tungsten Arc Welds

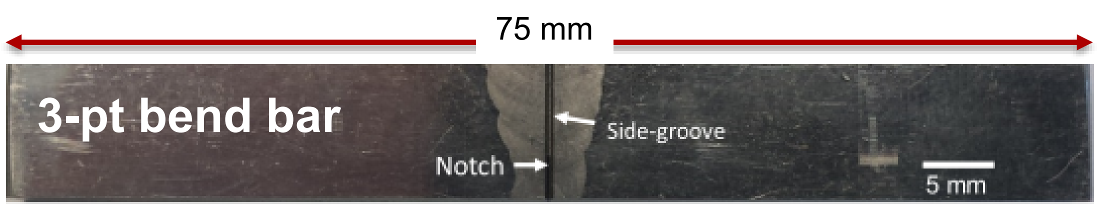
- Forgings were welded using same 308L filler metal

Material	Fe	Cr	Ni	Mn	Si	C	N	P	S	Yield Strength (MPa)
304L	Bal.	19.38	10.44	1.72	0.57	0.027	0.02	0.021	0.002	423
21-6-9	Bal.	21.21	7.16	9.21	0.51	0.029	0.28	0.016	0.0057	655
316L	Bal.	16.75	12.68	0.64	0.62	0.02	0.04	0.008	0.0023	482
308L Filler	Bal.	20.5	10.3	1.56	0.50	0.028	0.055	0.006	0.012	N/A

- J-grooves / Square-groove weld joint to permit testing in FZ or HAZ

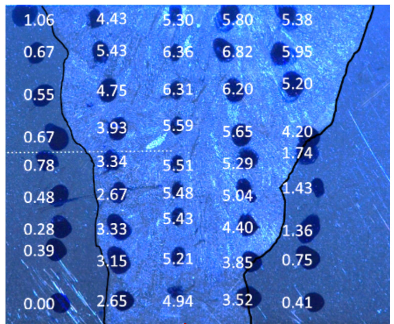


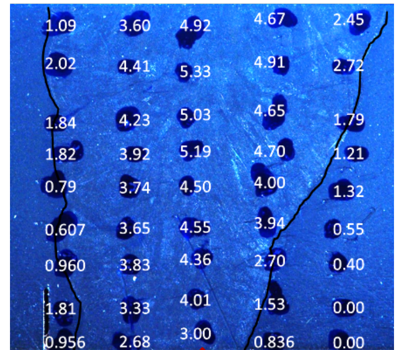
304L/308L 21-6-9/308L 316L/308L

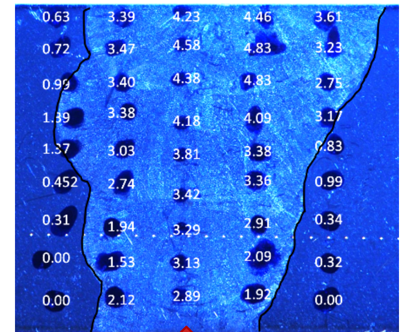


Ferrite content varied among welds fabricated with same filler metal

- Feritscope® was used to measure ferrite content in grid-like pattern



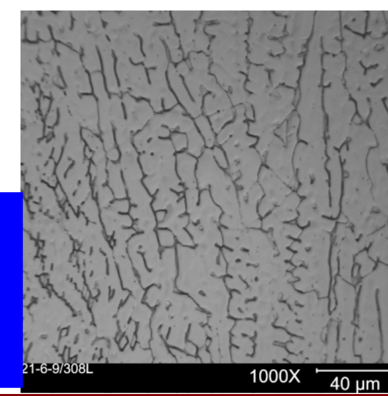




	304L / 308L	21-6-9 / 308L	316L / 308L
Avg. centerline ferrite content	5.6%	4.5%	3.8%

- Desirable to have primary δ-ferrite in order to prevent solidification cracking
- Despite same filler metal, dilution of 308L filler metal resulted in differences in ferrite content

δ-ferrite: black γ: grey

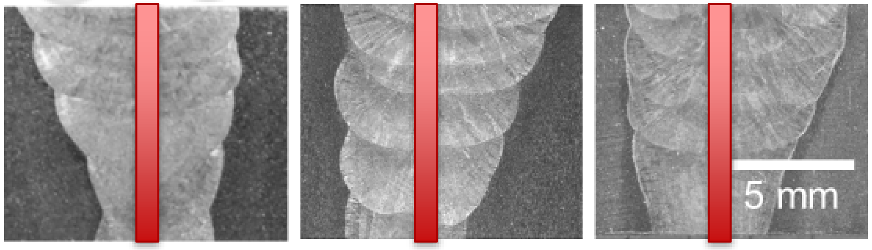


It is know that ferrite contributes to lower fracture toughness by acting as enhanced diffusion pathway, crack nucleation/coalescence site

Differences in Chemical composition between Base Metal and Weld

Base Metal Composition

Material	Fe	Cr	Ni	Mn	Si	C	N	P	S	Yield Strength (MPa)
304L	Bal.	19.38	10.44	1.72	0.57	0.027	0.02	0.021	0.002	423
21-6-9	Bal.	21.21	7.16	9.21	0.51	0.029	0.28	0.016	0.0057	655
316L	Bal.	16.75	12.68	0.64	0.62	0.02	0.04	0.008	0.0023	482
308L Filler	Bal.	20.5	10.3	1.56	0.50	0.028	0.055	0.006	0.012	N/A



Weld Centerline Composition

Material	Fe	Cr	Ni	Mn	Si	C	N	P
304L/308L	Bal.	19.8	10.1	1.66	0.43	0.026	0.04	0.03
21-6-9/308L	Bal.	20.3	8.42	5.9	0.5	0.027	0.11	0.03
316L/308L	Bal.	19.5	10.7	1.39	0.46	0.023	0.027	0.03

Dilution of weld metal from base metal may have an effect on deformation mechanisms: cross-slip versus planar slip

Weld tensile properties degraded at low temperature in H_2 precharged condition

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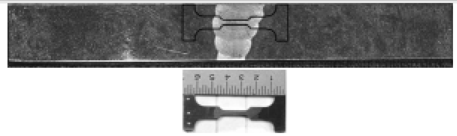
As-received 293 K

	Yield Strength (MPa)	Total Elongation (%)
304L/308L	289	59
21-6-9/308L	338	56
316L/308L	320	50

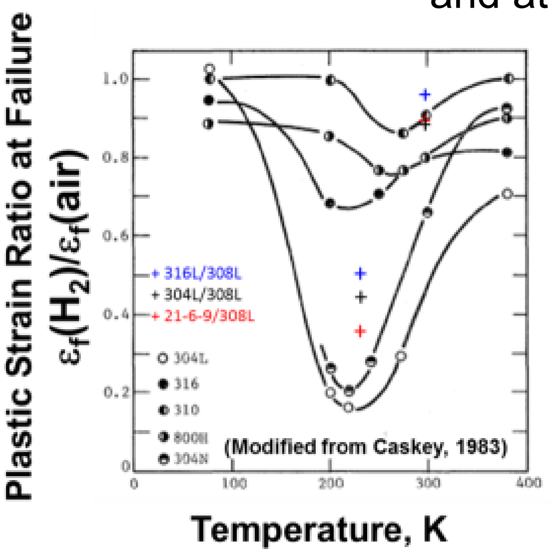


Hydrogen-precharged

	Yield Strength (MPa)		Total Elongation (%)	
	293 K	***223 K***	*293 K*	***223 K***
304L/308L	335	**341**	52	**25**
21-6-9/308L	430	**452**	50	**20**
316L/308L	337	**349**	49	**25**

*Test rate = 7.7 x 10^{-4} s^{-1}

- Yield strength increased in hydrogen-precharged condition and at low temperature



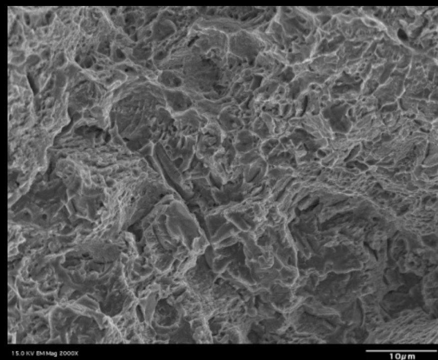
In Hydrogen Precharged Welds

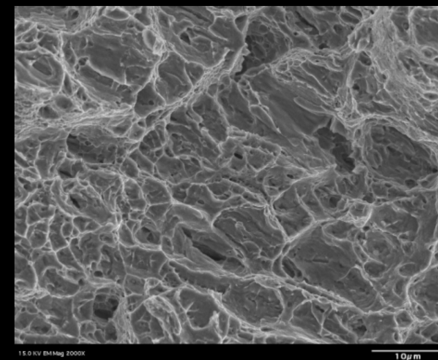
- Total Elongation decreased 3-11% at 293 K
- Total Elongation decreased 50-64% at 223 K
- Similar behavior to other 300 series stainless steels showing enhanced degradation at ~ 220 K

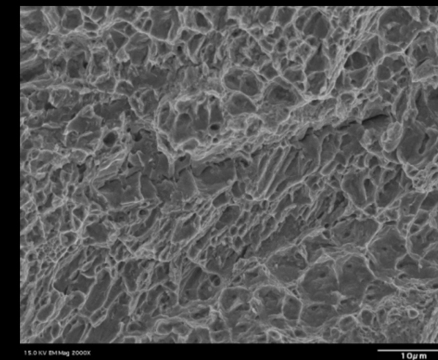
Hydrogen induced ductility loss was exacerbated at low temperatures

Greater microvoid coalescence in 293 K tests compared to 223 K weld tensile tests

293 K





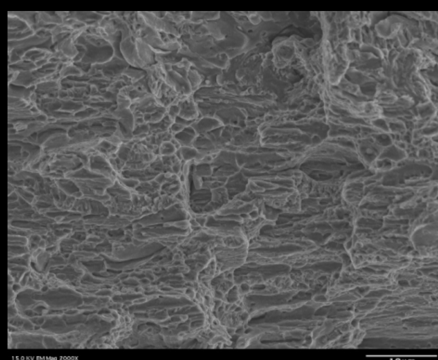


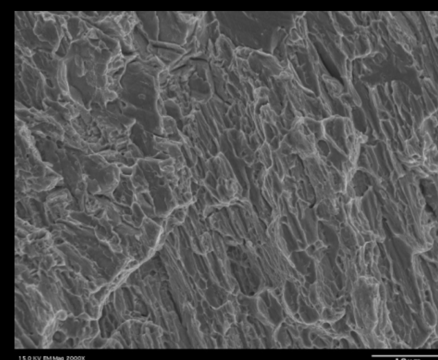
304L/308L

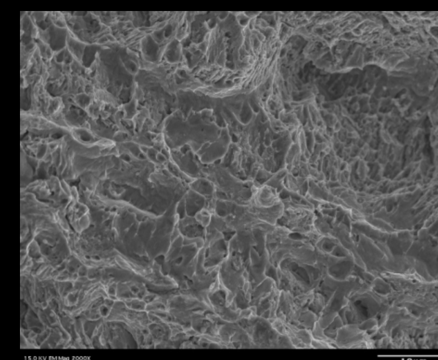
21-6-9/308L

316L/308L

223 K







Greater loss in ductility at low temperature results in more brittle fracture features

Fracture Threshold (J-R curves) measured on hydrogen-precharged at 293 K and 223 K

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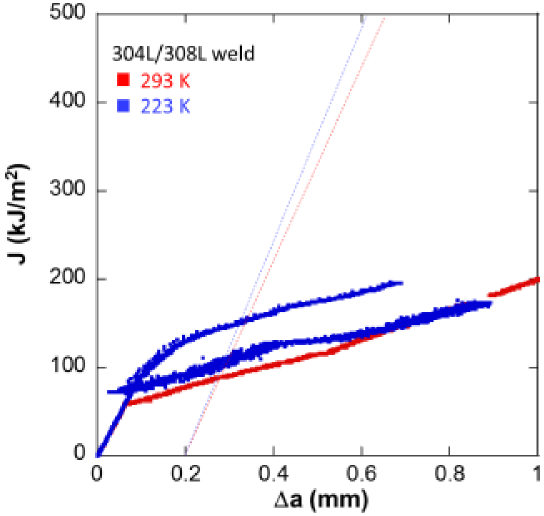
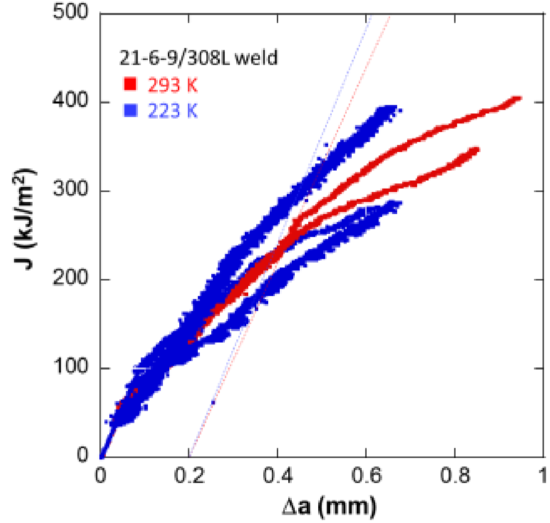
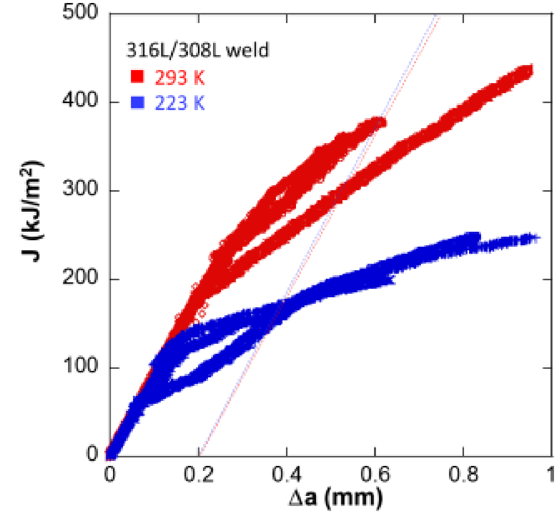


304L/308L



21-6-9/308L



316L/308L

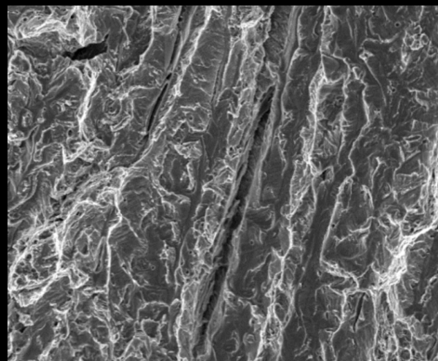
Test rate 0.02 mm/min
(0.1 MPa $m^{1/2}/s$)

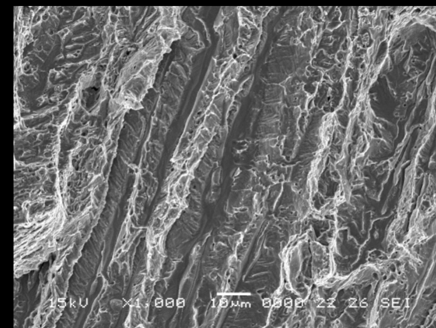
- In non-charged condition, negligible crack extension observed
- 304L/308L weld exhibited lowest fracture thresholds of three welds
- 316L/308L weld exhibited significant degradation at low temperature

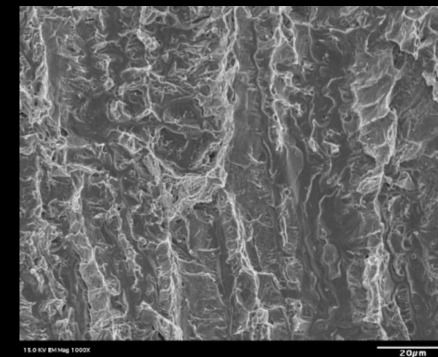
Temperature dependence only observed in 316L/308L weld fracture behavior

Fracture tests fractography revealed distinct differences at 293 K and 223 K

293 K





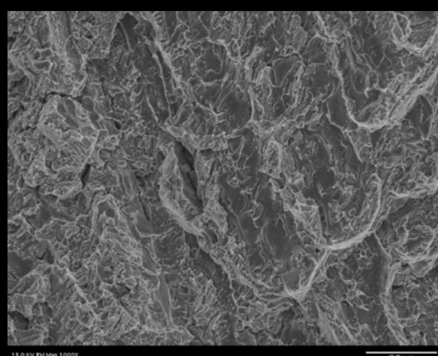


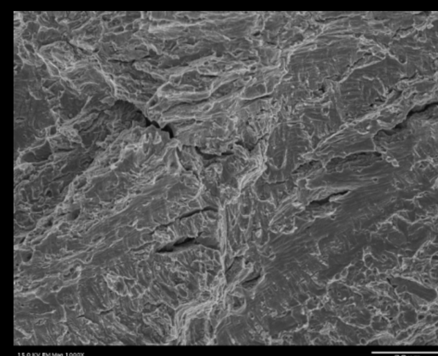
304L/308L

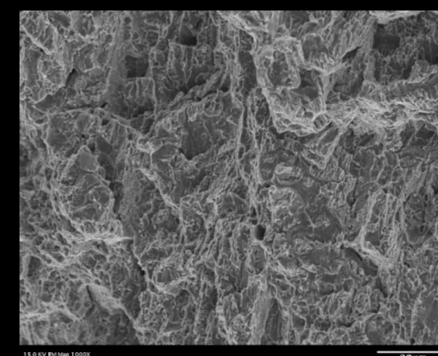
21-6-9/308L

316L/308L

223 K



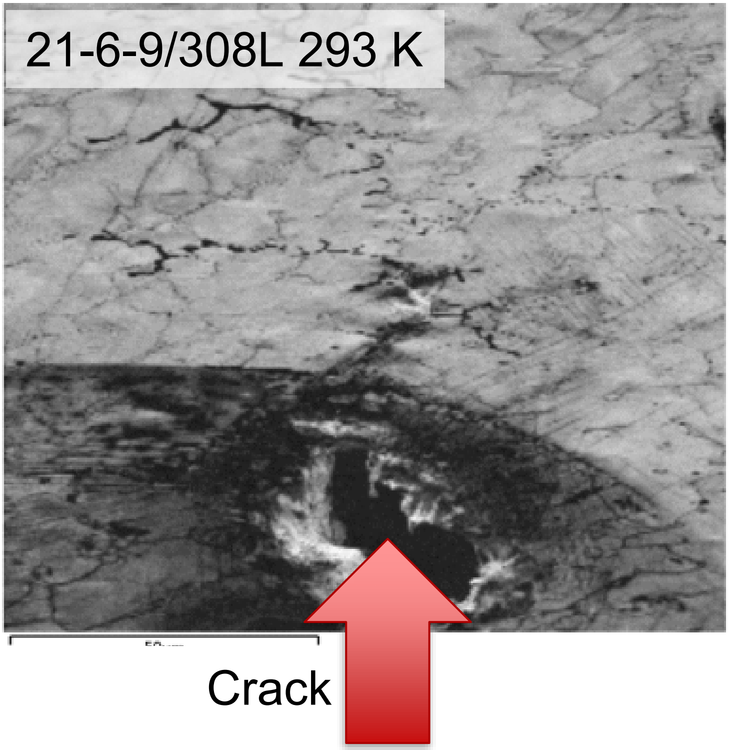




- At 293 K, dendritic structure was visible on weld fracture surfaces
  - Clear that dendritic ferrite participated in crack nucleation & growth
- At 223 K, dendritic structure mostly absent

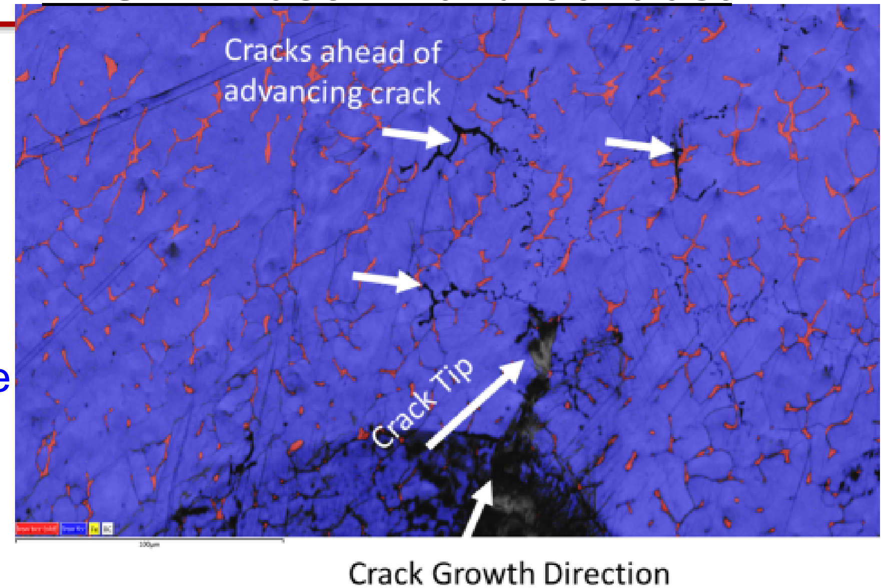
Differences in fracture surface features suggests ferrite participates differently in fracture at low temperature

Deformation structures ahead of crack tip at 293K

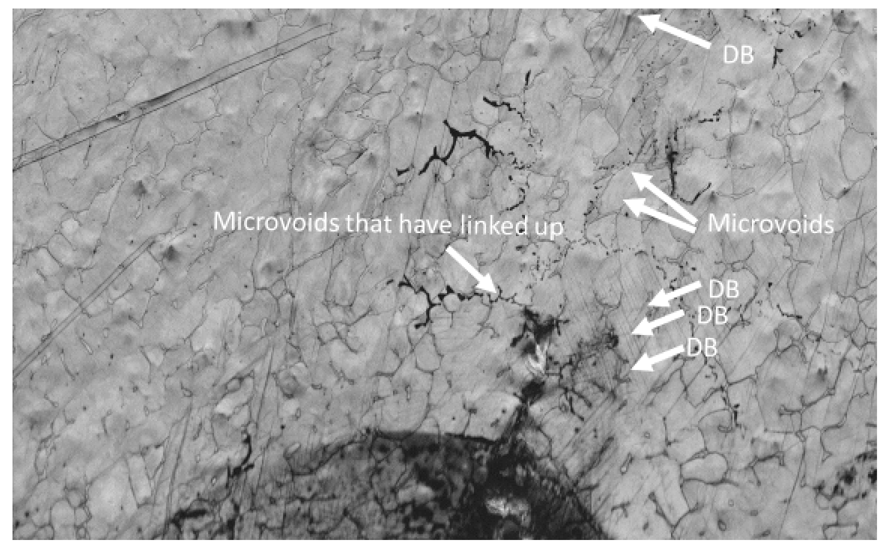


Ferrite: red
Austenite: blue

EBSD: Phase + Band Contrast



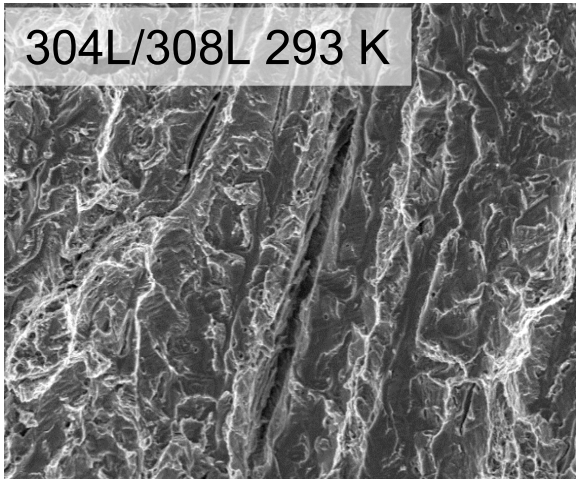
EBSD: Band Contrast



293 K

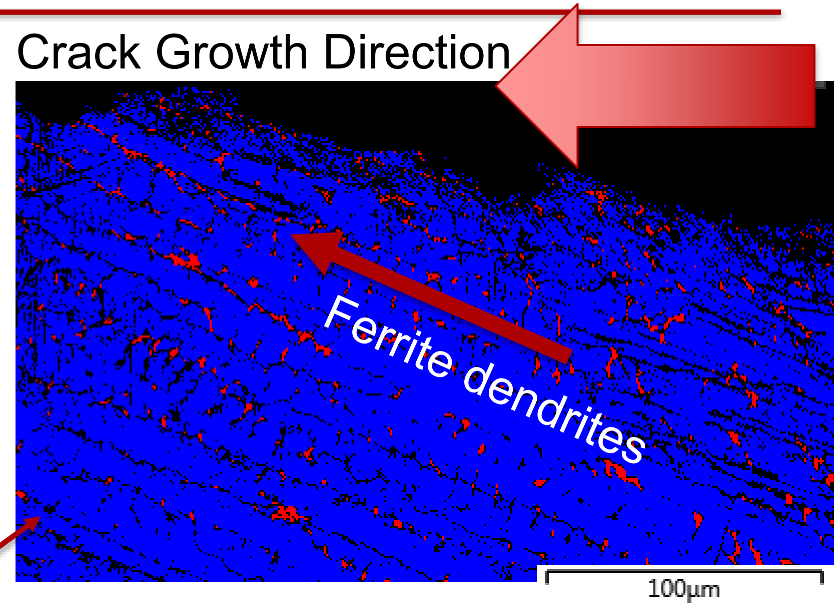
- Damage occurs as voids which link up to form microcracks.
- Deformation bands (DB) appear ahead of advancing crack
- Damage located at or near ferrite

Deformation structures below fracture surface at 293K



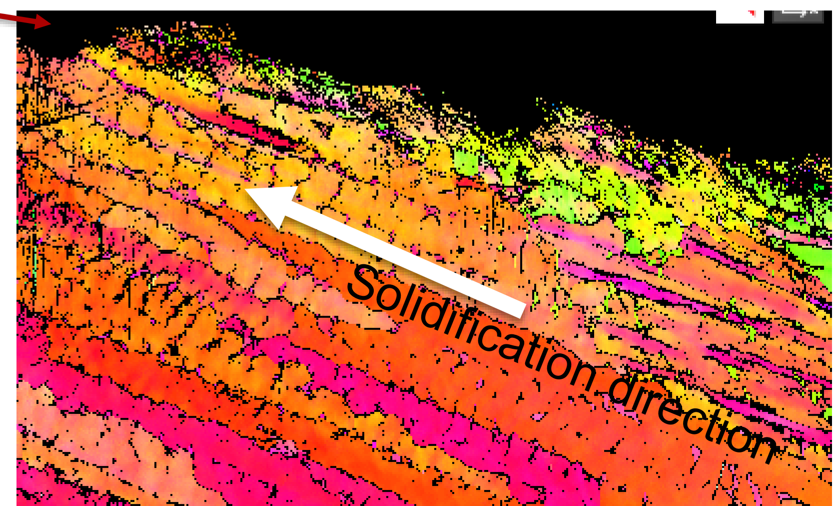
Crack Growth Direction

Ferrite: red
Austenite: blue



EBSD: Phase

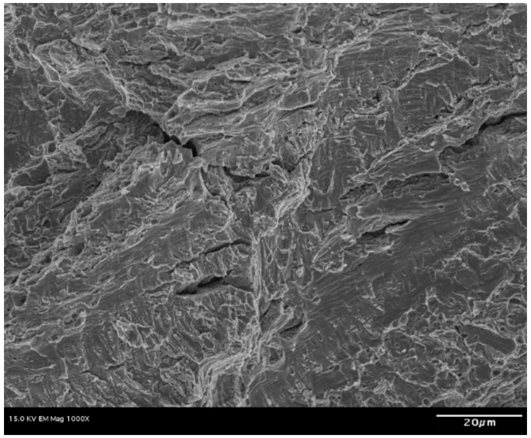
IPF

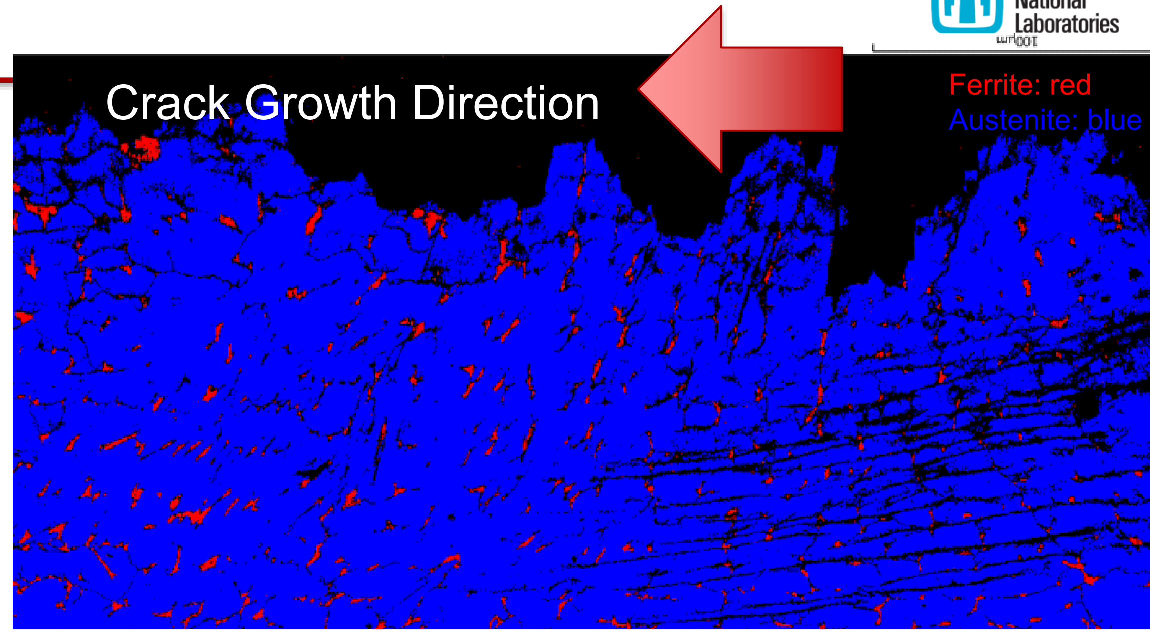


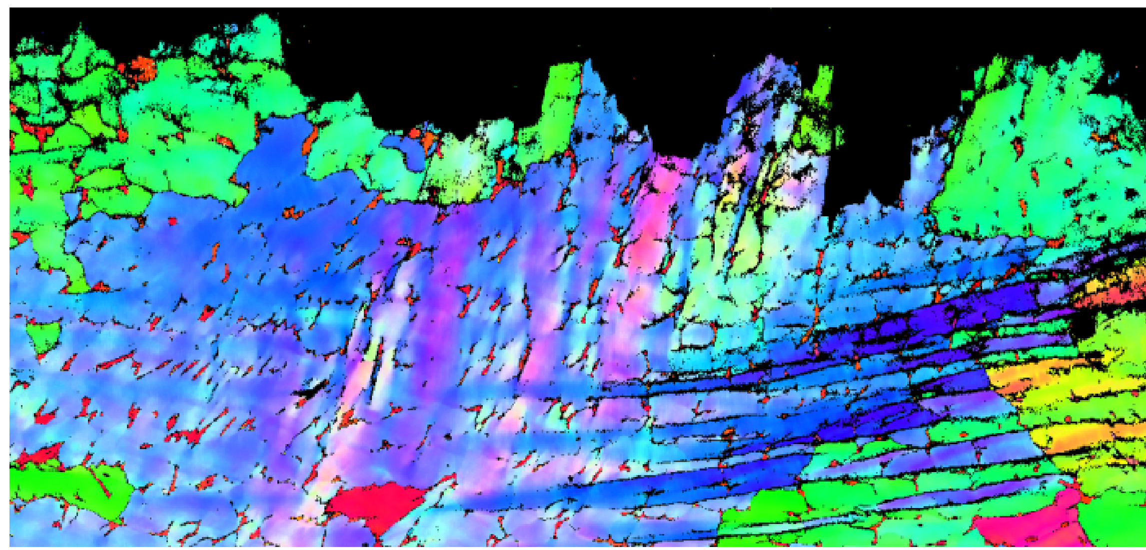
- Fracture path follows dendritic structures
- Ferrite appears to be predominant pathway.

Deformation structure below fracture surface at 223 K

21-6-9/308L 223 K

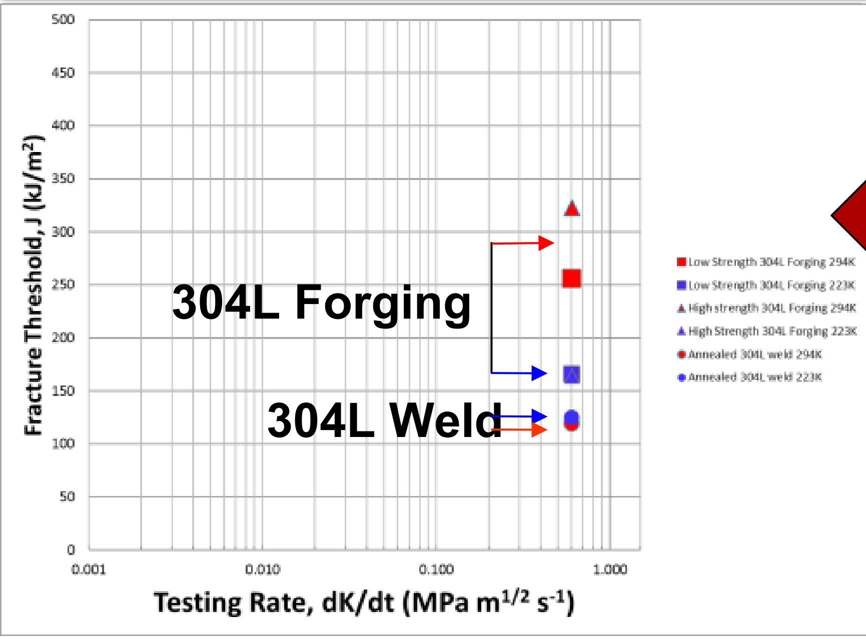






- Dendritic features are absent on fracture surface
- Crack profile is more tortuous, e.g. does not follow solidification dendrites

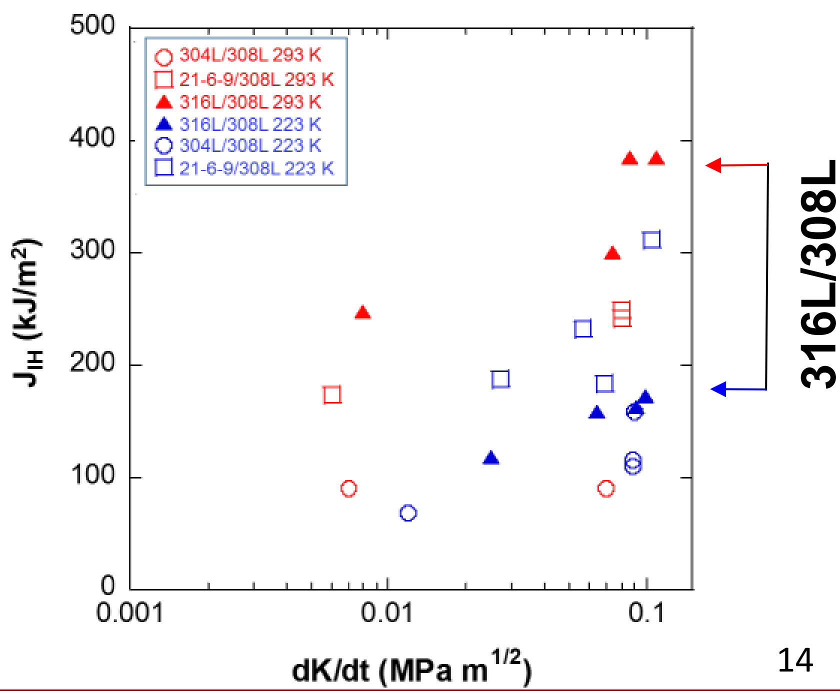
Temperature Effects on Forgings vs Welds



Jackson *et al* (2012,2016)

- 304L Forgings exhibited temperature effects
  - **Low T = lower toughness**
- Annealed 304L/308L welds → No temp effects

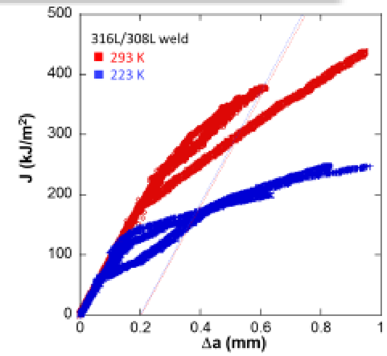
- Minimal temperature effects on fracture toughness for 304L and 21-6-9 welds
- 316L welds → Over 50% decrease in toughness for 316L welds at **223 K**
- Similar rate dependence at both temperatures

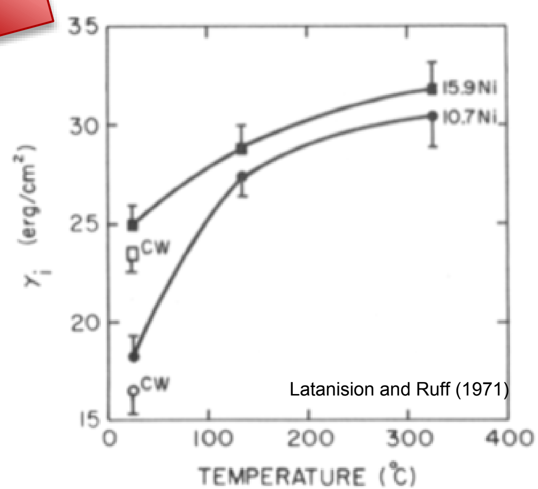


Low Temperature can exacerbate slip planarity

316L/308L weld exhibited enhanced degradation at low temperature

- Low temperature reduces SFE and enhances planar slip
- High Ni-content promoted cross slip at 293K
    - At 223 K, deformation may transition into planar slip accounting for large reduction in toughness

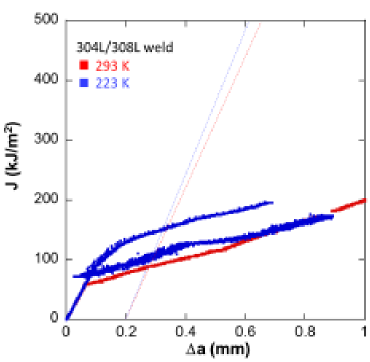


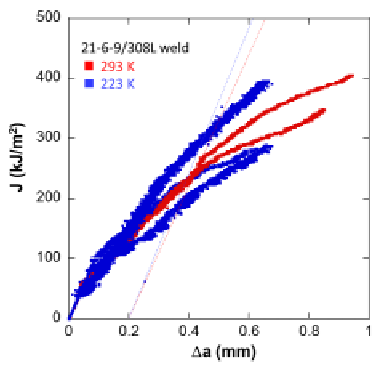


Latanision and Ruff (1971)

304L/308L and ***21-6-9/308L*** welds contain lower Ni-content and therefore may already exhibit a significant amount of planar slip at 293 K

- Therefore less sensitive to temperature when tested at 223 K.





Composition, Temperature, and hydrogen can influence deformation mechanisms

Summary

- Fracture tests and tensile tests were performed on 3 austenitic stainless steel welds in hydrogen-precharged condition
  - Modest reductions in tensile ductility (3-11%) were measured at 293 K, whereas ductility losses of 50-64% were observed at 223 K
  - Fracture thresholds were greatly reduced at all test temperatures
    - 304L/308L and 21-6-9/308L exhibited negligible temperature dependence
    - 316L/308L exhibited over 50% decrease in fracture thresholds at 223 K vs 293 K
- Examination of fracture surfaces suggested deformation evolved differently at low temperatures
  - Fracture followed dendritic ferrite regions at 293 K
  - More tortuous pathways were observed at 223 K
- Temperature, Chemical composition, and hydrogen can alter deformation mechanisms
  - Cross slip → Planar slip resulting in more enhanced degradation

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jaronev@sandia.gov