SAND2021-11015C

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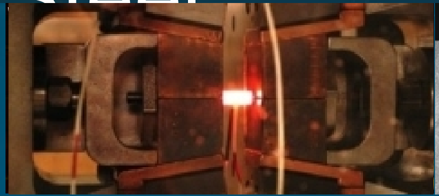
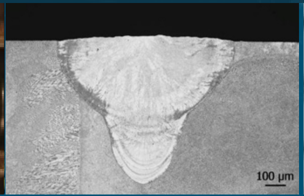
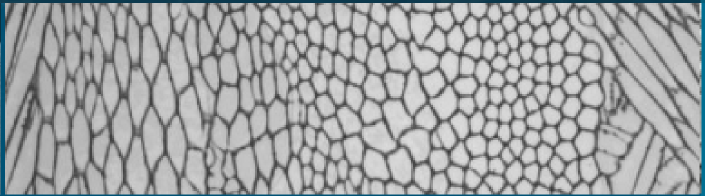
Microstructural and Mechanical Analysis of Intermediate Temperature Equilibrium Phase Formation in Nitronic 60 Stainless Steel







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Bellows assemblies often are complex multi-alloy designs

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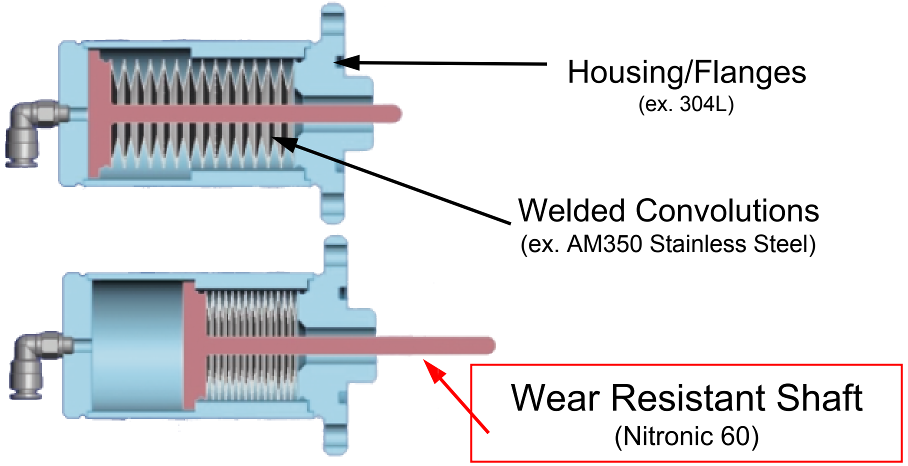
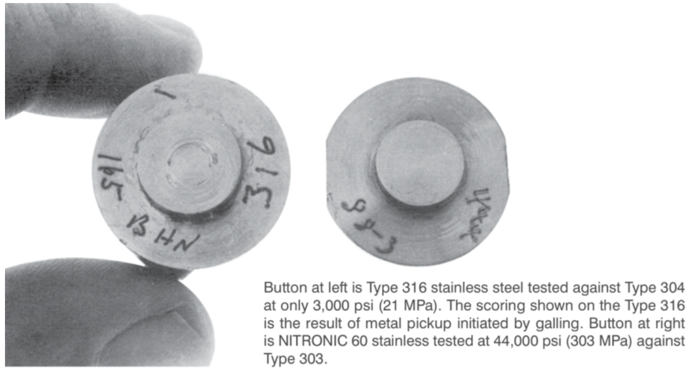
- Metal bellows are a flexible metal connection that separates two mechanical environments while allowing linear or rotational motion.
- Bellows assemblies that can be used for actuation between two environments are typically multi-alloy systems fabricated in multiple steps
- Galling resistant stainless steels like Nitronic 60 are attractive alternatives to 304L and offer excellent corrosion resistance, toughness, and tribological properties

Generalized Schematic of a Bellows Assembly

(assembly example shown from Senior Aerospace[1])





From High Performance Alloys Nitronic 60 Product Brochure[2]

Specified ASTM Composition Ranges for N60 and 304L

Element	304L (ASTM A240)	Nitronic 60 (ASTM A240/A276)
C	0.030 max.	**0.10 max.**
Mn	2.00 max	**7.0 - 9.0**
P	0.040 max.	0.040 max.
S	0.030 max.	0.030 max.
Si	0.75 max.	**3.50 - 4.50**
Cr	17.5 - 19.5	16.0 - 18.0
Ni	8.0 - 12.0	8.0 - 9.0
N	0.10 max.	**0.08-0.18**
Nb	*Not Specified*	*Not Specified*
Fe	bal.	bal.

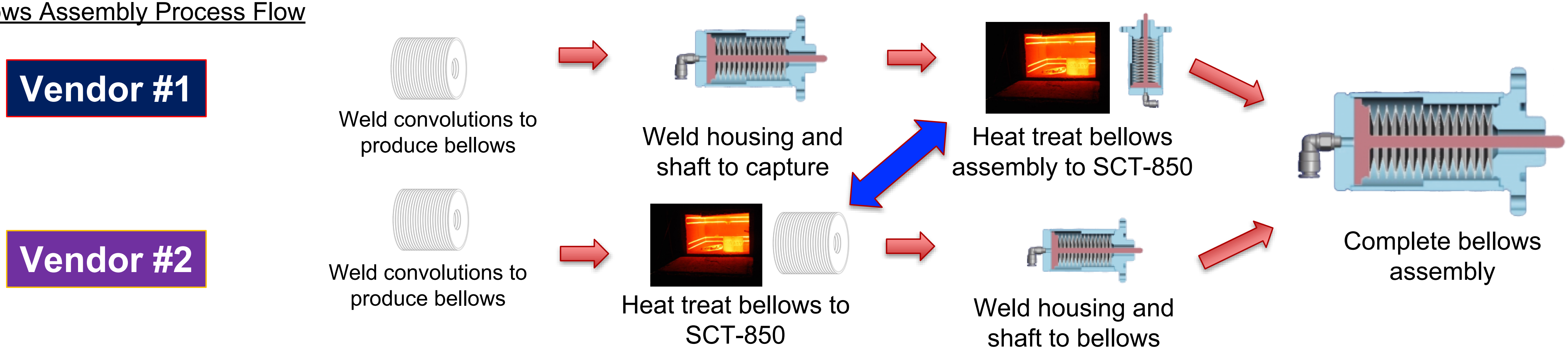
Elements in red are alloying additions to N60 to alter: yield strength, stacking fault energy, and post-yield deformation mechanisms to improve galling resistance

[1]Senior Aerospace: https://www.metalbellows.com/assets/BellowsDevicesforVacuum.pdf
[2]High Performance Alloys: https://www.hpalloy.com/Alloys/descriptions/NITRONIC60.aspx

Welded Bellows Case Study: One design, two methods of assembly

- Two approved vendors manufacture bellows assemblies with Nitronic 60 shafts to the same design
- Welded bellows produced from precipitation-strengthened AM350 stainless steel requires heat treatment
- To make the assemblies per the drawing, the exact order of manufacturing steps varies between Vendor #1 and Vendor #2

<u>Bellows Assembly Process Flow</u>



Upon receipt of parts, testing of N60 tensile witness samples made at Vendor #1 indicates anomalously low ductility

- Tensile testing of N60 witness samples from part lots 1 and 2 revealed non-conforming ductility values (both elongation-to-failure and reduction in area)
  - Specification requirements %elongation and %reduction in area values: 35% min. and 55% min., respectively
  - *Typical values: ~60% and ~65%, respectively*

- Originally a testing error was suspected given the magnitude of reported ductility values

- Chemical analyses were performed to confirm tensile specimens were consistent with N60

Reported ductility values sufficiently low to call into question the mechanical integrity of the bellows shaft

Reported tensile data for N60 samples from Lot 1 and Lot 2

Lot 1 samples 1, 2, and 6 examined

		Lot 1						Lot 2		
		Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 1	Sample 2	Sample 3
(req. min. 35%)	Elongation (%)	12.9	13.2	21.5	16.9	10.8	24.4	24.4	0	15.6
(req. min. 55%)	Reduction in Area (%)	20.4	9.6	20	26.7	15.2	23.3	23.1	0	17.8
	Yield Strength, 0.2% (ksi)	54.6	54.7	54.7	55.1	55.1	54.7	55.4	55.9	55.1

Bulk Chemical Analysis of Low Ductility Tensile Samples

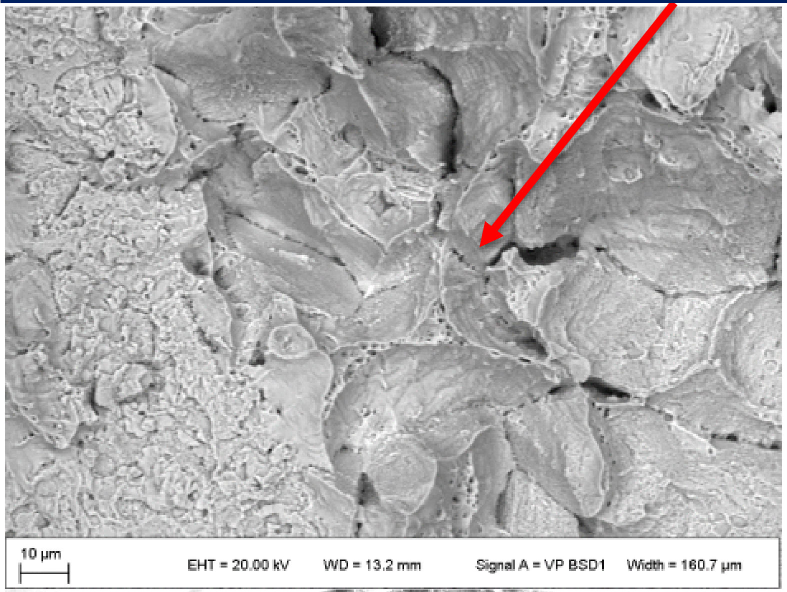
- One half of each fracture surface was used for digestion-based measurement (total of three samples co-digested)
  - Fracture surfaces were removed before digestion and preserved
- Bulk alloy chemistry does not reveal any deleterious levels of unspecified elements. Moreover, chemistry values are in good agreement with materials certification sheet
- <u>Alloy would meet ASTM A240/A276 composition requirements for N60. Measured concentrations of unspecified elements are unremarkable.</u>

	wt%			
Element	Bulk Analysis Lot 1 Samples 1,2,6	Measurement Technique	Material Certification	ASTM A240/A276
C	0.075	LECO	0.08	0.10 max
Mn	8.38	ICP	8.70	7.00-9.00
P	0.019	ICP-MS	0.020	0.040 max.
S	<0.001	LECO	<0.001	0.030 max.
Si	3.98	ICP	3.99	3.50-4.50
Cr	16.50	ICP	16.55	16.00-18.00
Ni	8.07	ICP	8.38	8.00-9.00
N	0.14	IGF	0.14	0.08-0.18
Al	0.006	ICP-MS	*Not Evaluated*	*Not Specified*
Co	0.060	ICP-MS		
Cu	0.092	ICP-MS		
Mg	<0.001	ICP-MS		
Mo	0.29	ICP-MS		
Nb	0.058	ICP-MS		
O	0.022	IGF		
Sn	0.003	ICP-MS		
Ti	0.018	ICP-MS		

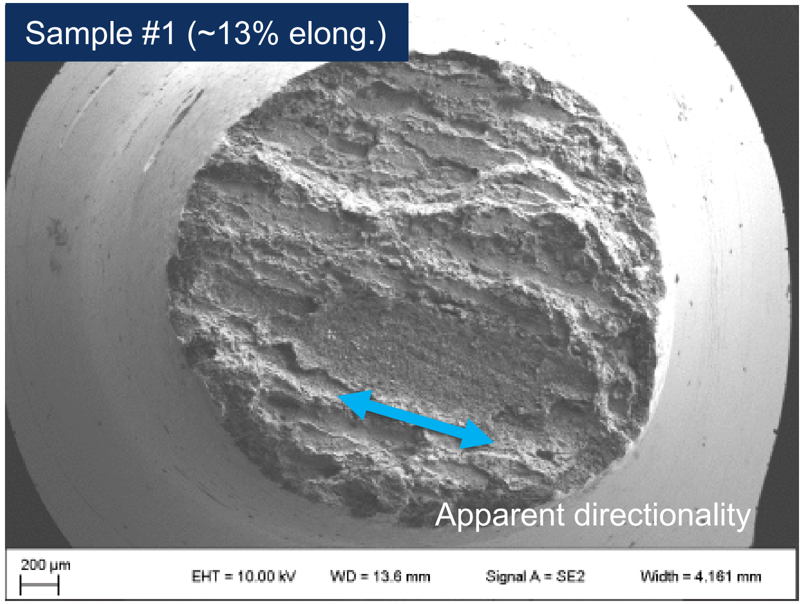
Fractographic examination of tensile samples shows brittle features and unexpected directionality

- Fractographic examination shows flat fracture (associated with low-ductility) with visible directionality on fracture surface
- No expected 'cup-cone' fracture morphology observed
- High-magnification examination shows intergranular fracture

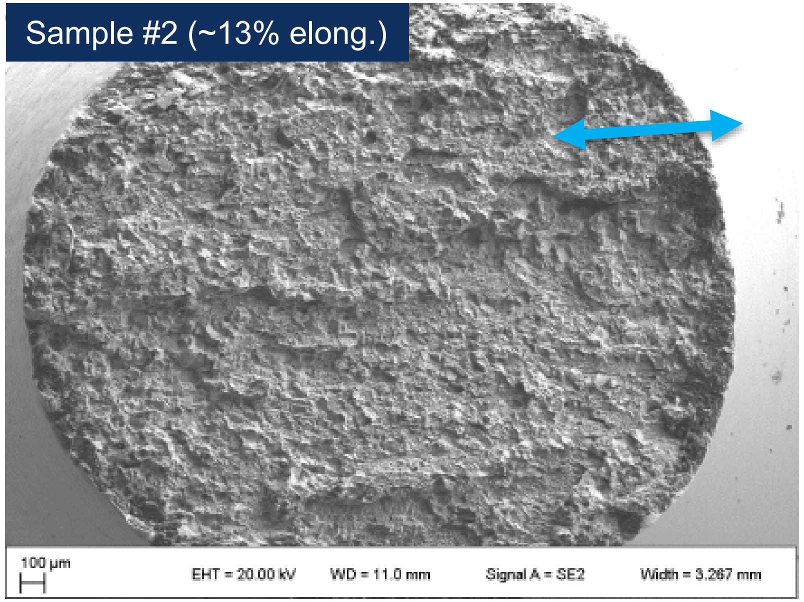
Grain boundary second phase observed in intergranular fracture



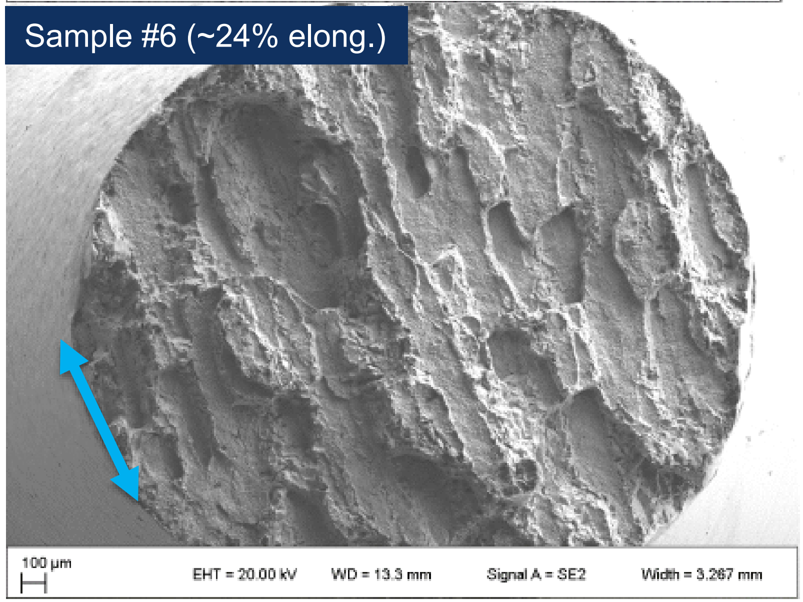
Sample #1 (~13% elong.)



Sample #2 (~13% elong.)



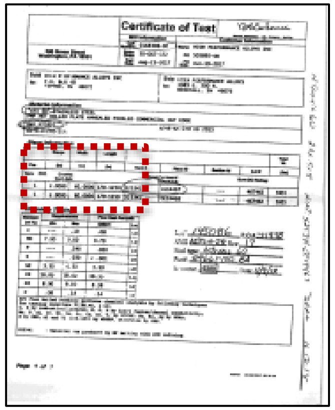
Sample #6 (~24% elong.)

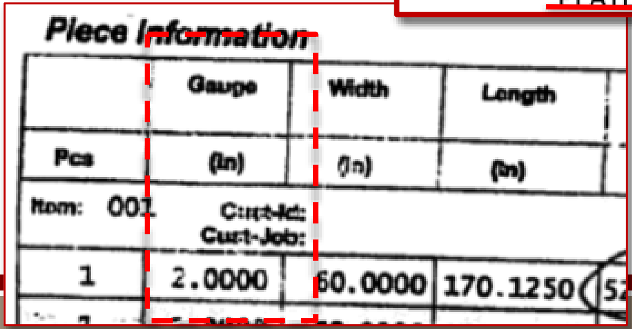


Directionality Explained: tensile samples and bellows shaft parts made at Vendor #1 were machined from plate short transverse (thickness) direction

- Vendor #1 elects to made the bellows shaft from the alternate material callout on the drawing—N60 plate per ASTM A240. Vendor also confirmed that parts are from plate thickness direction!
- For plate, tensile properties are expected to be the lowest along the short transverse direction due to grain orientation, macrosegregation, and aligned second-phases
- For N60 thick plate, changing from long to short transverse orientation results in ~33% reduction in ductility.

N60 Cert Parts Made by Vendor #1





Piece Information

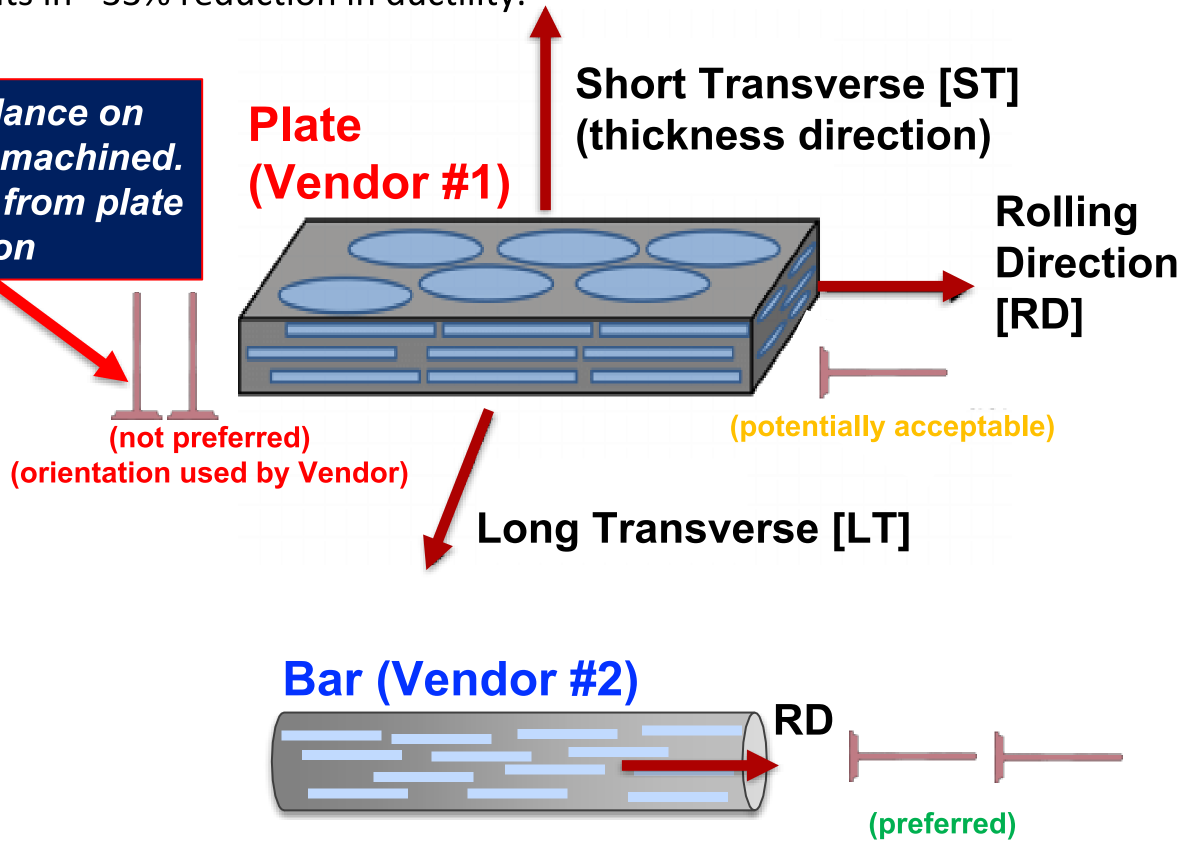
	Gauge	Width	Length	
Pcs	(in)	(in)	(in)	
Item: 001	Cust-Id: Cust-Job:			
1	2.0000	60.0000	170.1250	(52

Shaft drawing gives no guidance on which direction parts shall be machined. Vendor #1 elected to machine from plate in unfavorable direction

Bellows Shaft Drawing Note

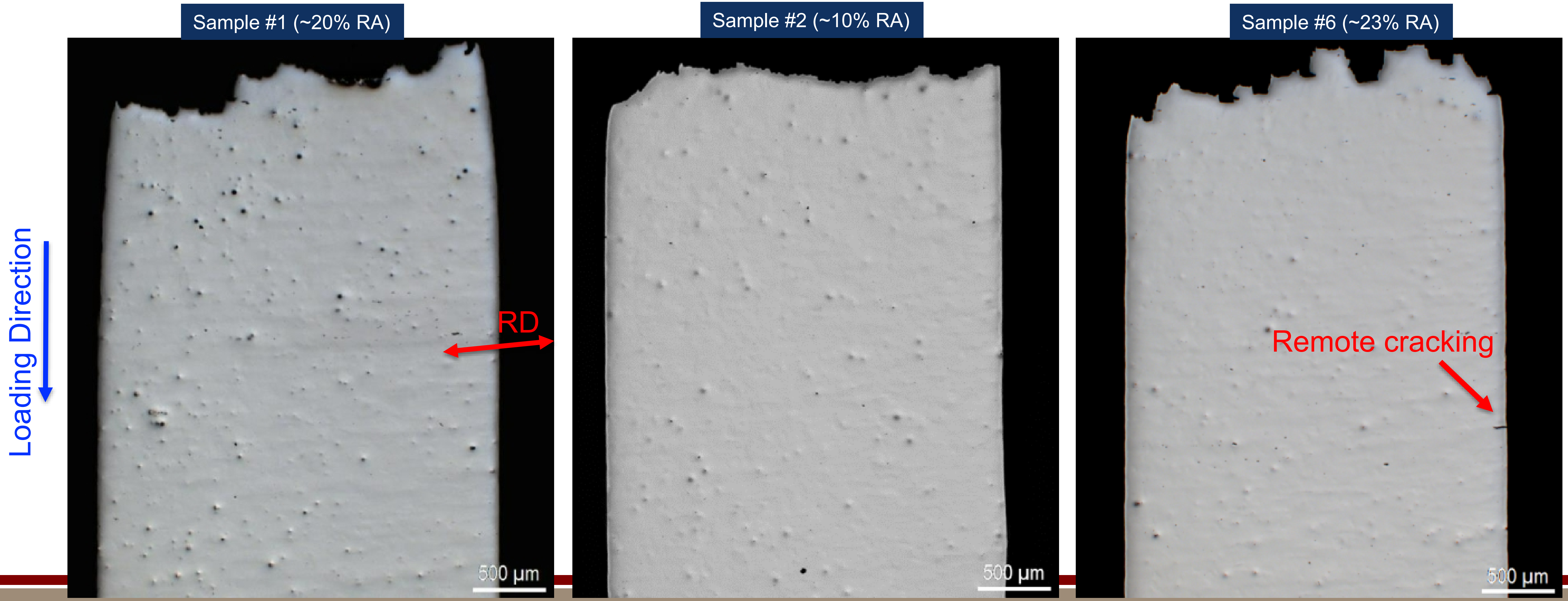
3. MATERIAL:
   A. STAINLESS STEEL ALLOY UNS 21800 (NITRONIC 60), BAR (ANNEALED) PER ASTM A276 OR UNS 21800 (NITRONIC 60) PLATE PER ASTM A240

N60 plate is a permissible alternate material form according to the drawing



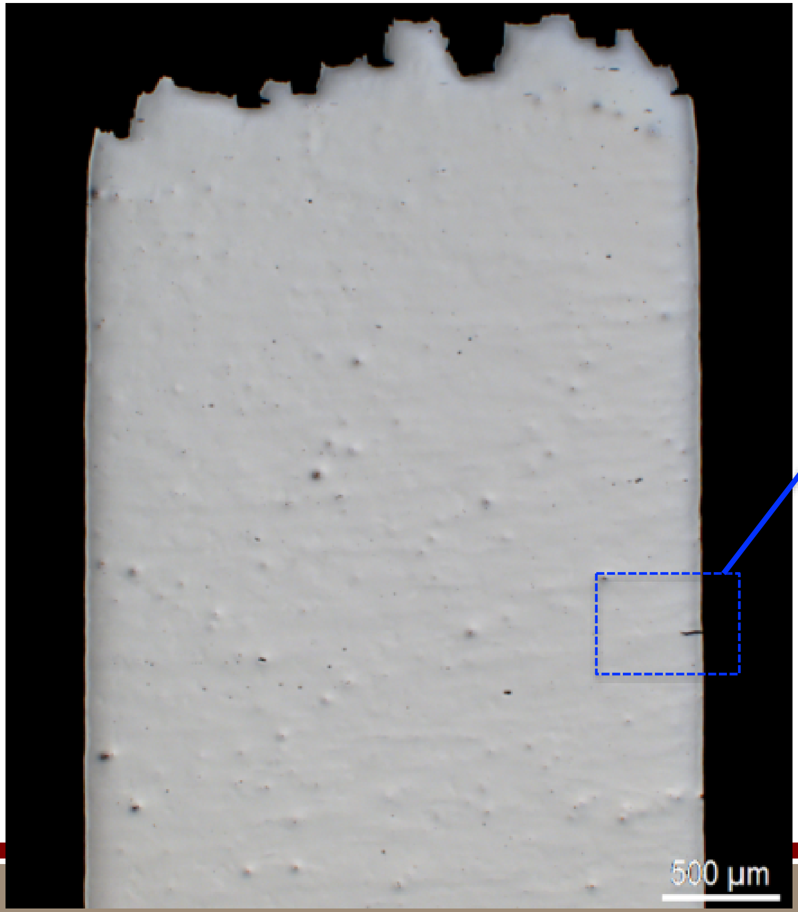
Short transverse direction alone will not result in the magnitude of ductility nonconformance observed

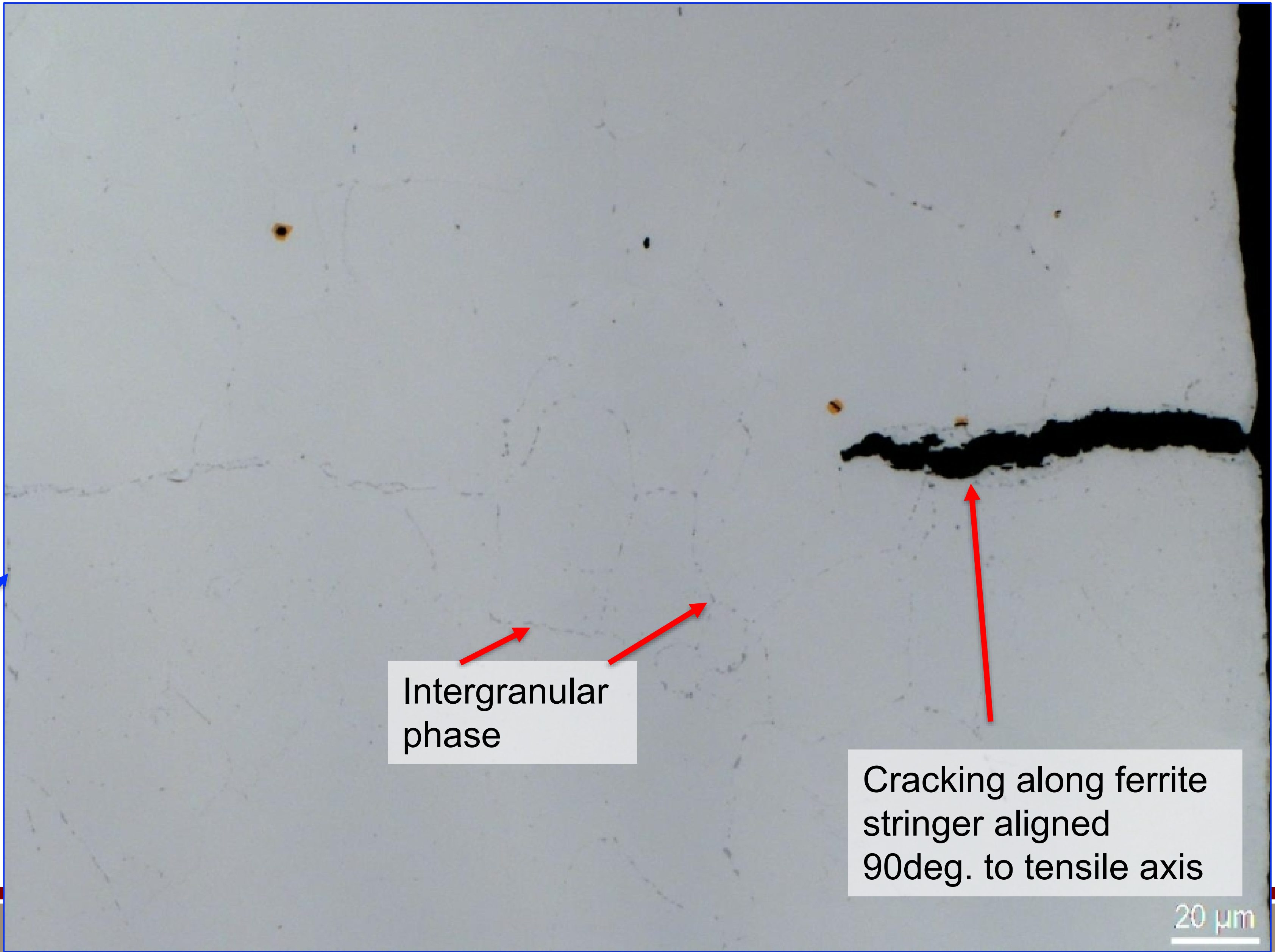
- Cross sections reveals relatively low extent of necking (i.e., low ductility/reduction in area) near fracture.
- Cracking remote from final fracture surface was observed for sample #6. This behavior is consistent with embrittled materials.
- Brittle cracking along ferrite stringers and grain boundaries is not expected behavior for austenitic stainless steels

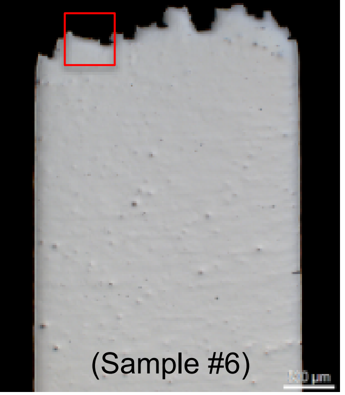


Sample #6: Cracking observed remote from fracture

- Cracking in tensile specimen remote from fracture surface is indicative of operative embrittlement

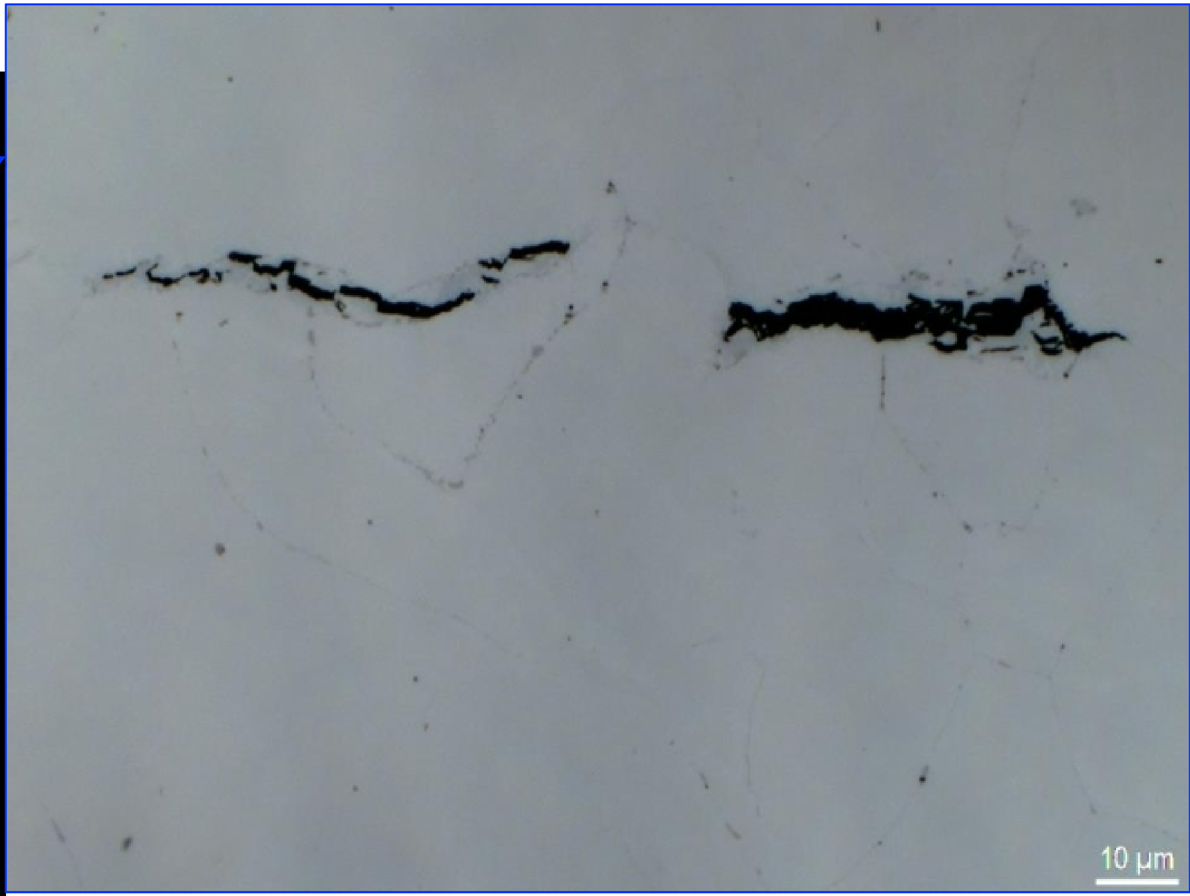






Sample #6: Cracking observed within ferrite phase oriented along plate rolling direction

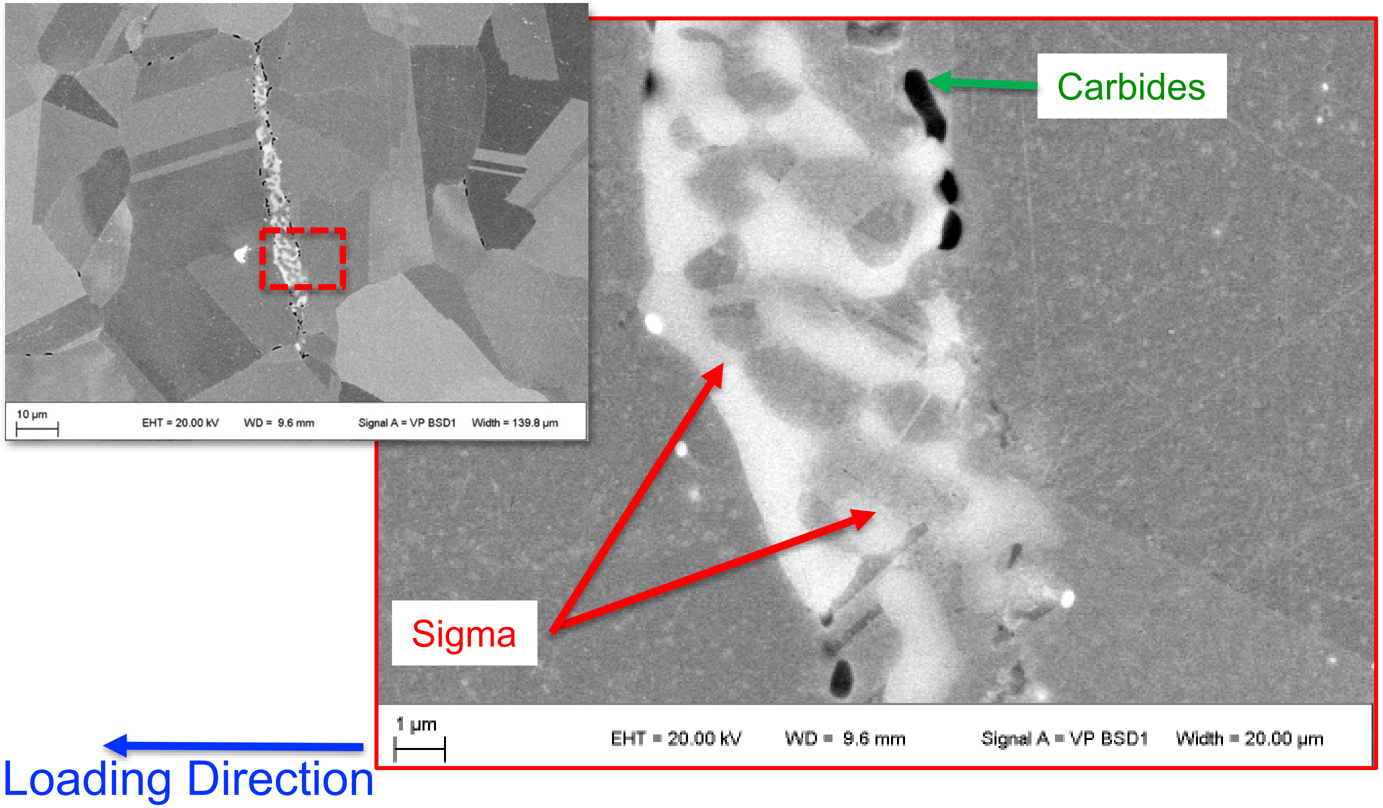
- Fracture observed remote from fracture surface in ferrite phase aligned with plate rolling direction.



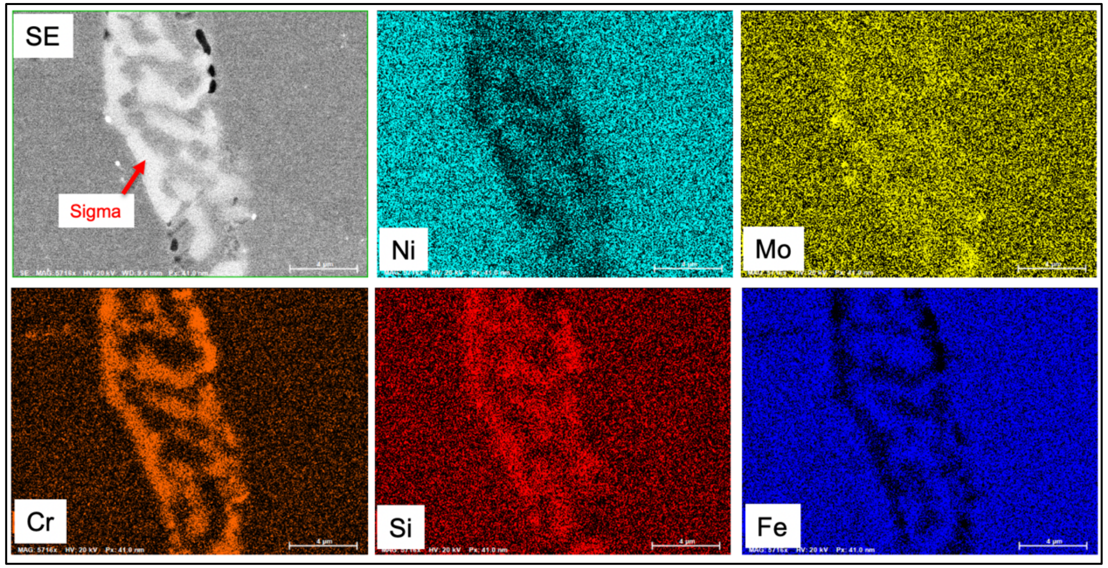
Cracking in stringer phase. Intergranular phase formation also visible

Detailed microstructural analysis confirms that N60 tensile samples exhibit sigma phase embrittlement

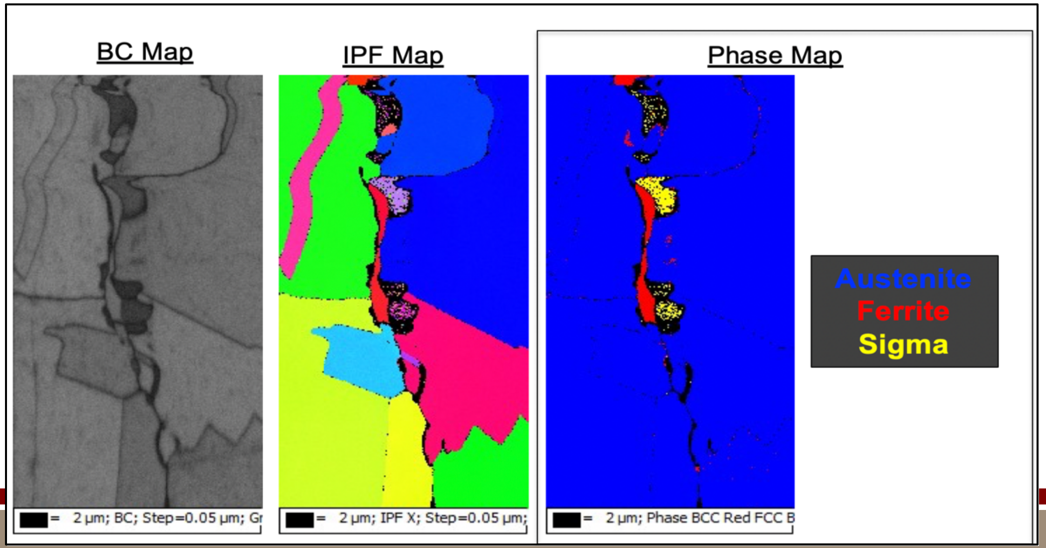
- Low-ductility bellows N60 shaft tensile witness coupons show sigma phase throughout microstructure where ferrite stringers would be normally
- Sigma phase was identified based on morphology, chemistry, and crystal structure



Sigma phase identified compositionally (EDS)



Sigma phase identified structurally (EBSD)



Additional comments about sigma phase in stainless steels

- Very limited data exists for sigma phase formation in N60
- Sigma phase is a low-temperature equilibrium phase that forms in stainless steels
- Sigma is hard, brittle and generally deleterious to the mechanical behavior and corrosion resistance
- Sigma phase is generally sluggish to form (many hours of exposure between ~600-800C); <u>however, sigma phase can form much more quickly if delta-ferrite is present</u>
  - Delta-Ferrite → Sigma + Austenite
  - Elevated Si content of N60 likely enhances kinetics relative to 304L

<u>Kinetics of sigma formation in N50 are rapid when delta-ferrite is present</u>

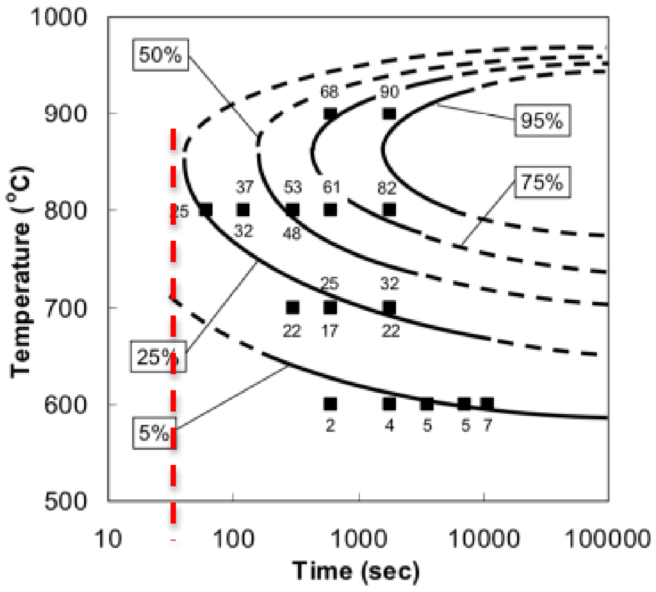
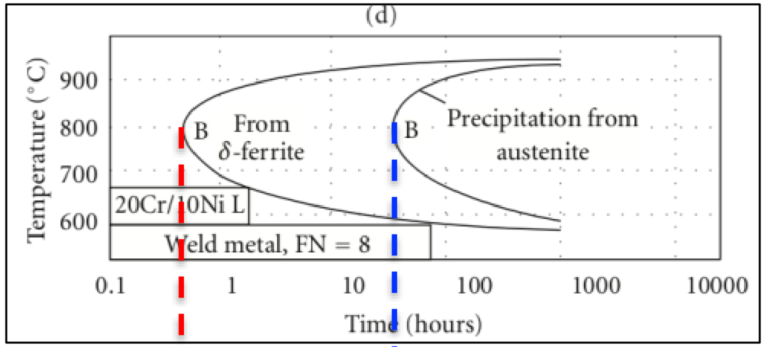


Figure 7. Time – temperature – transformation diagram for the delta ferrite to sigma phase transformation. Black squares are calculated amounts of transformation (with some duplicates at 700 and 800°C). 5 to 95% transformation contours were calculated from linear least-squares fits of the transformation vs. log time data at each temperature.

(from Balch et al., Proc. TWR, 2005.)

<u>Sigma formation kinetics from ferrite vs. from austenite in 20Cr10Ni stainless weld metal</u>



(from E. Folkhard, Welding Metallurgy of Stainless Steels, Springer, New York, NY, USA, 1st edition, 1988)

Onset of sigma phase formation can occur ~2 orders of magnitude shorter duration of ferrite is present

Recall: Vendor #1 heat treats the entire bellows assembly to SCT-850

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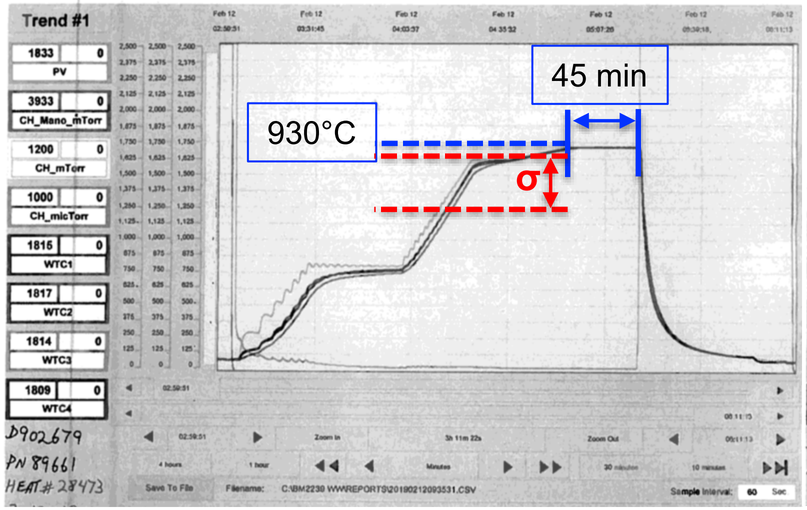
- The heat treatment used to generate desirable properties of AM350 convolutions results in N60 shaft exposed to temperatures favorable for sigma embrittlement
- Sigma forming during heat treat results in up to 80% reduction in ductility

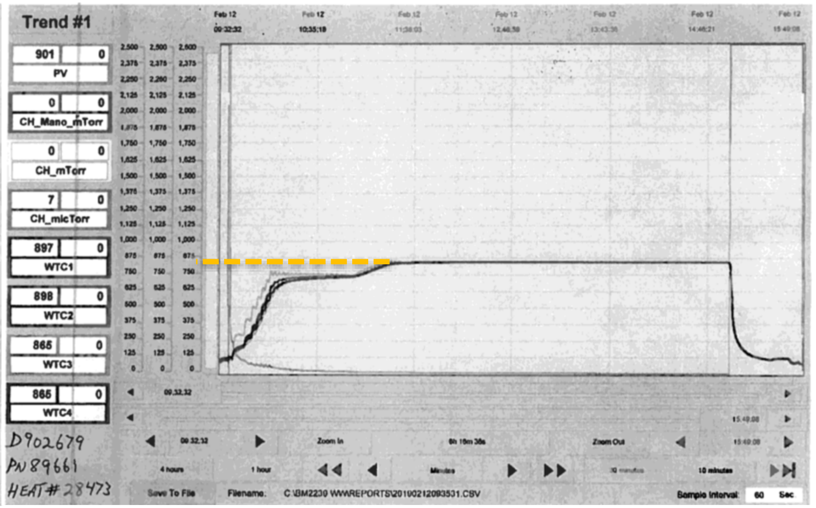
SCT 850

- 930°C, 45 mins
- Cool rapidly to -73°C 1-3 hours
- 450°C, 3 hours

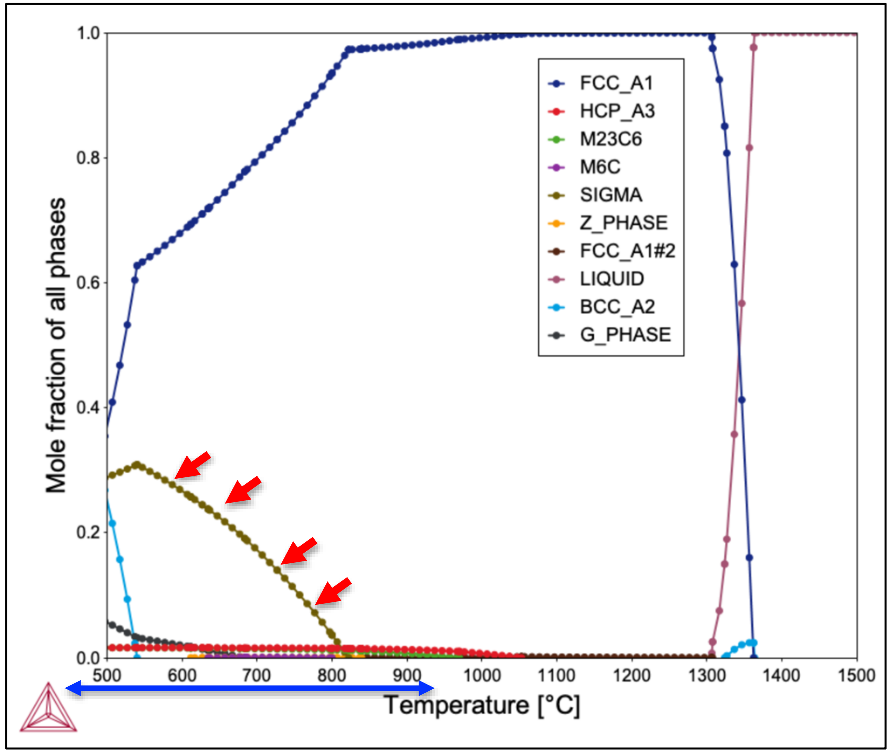
SCT-850: austenite conditioning



SCT-850: aging



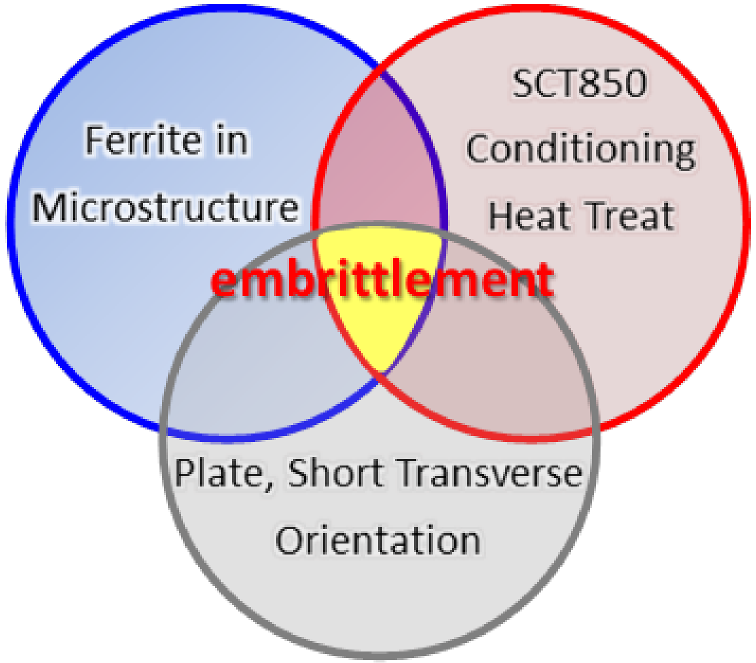
Multicomponent thermodynamic prediction of phase content vs. temperature for N60



Embrittlement of bellows assembly shafts is the result of a 'perfect storm' of processing conditions

- <u>Elimination of any one of these factors would likely not result in diminished N60 ductility from sigma</u>
- Without additional testing, it is difficult to assert the assemblies will meet design performance requirements given the extent of sigma phase embrittlement

Ferrite content in N60 can vary and is not controlled. The 2.0" plate used for shafts demonstrating low ductility contained ~ 1 vol. % ferrite



Vendor #1 applies SCT-850 to the entire bellows assembly (including N60 shaft)

The drawing allowed N60 plate as an alternate with no guidance on acceptable orientation of samples and/or parts

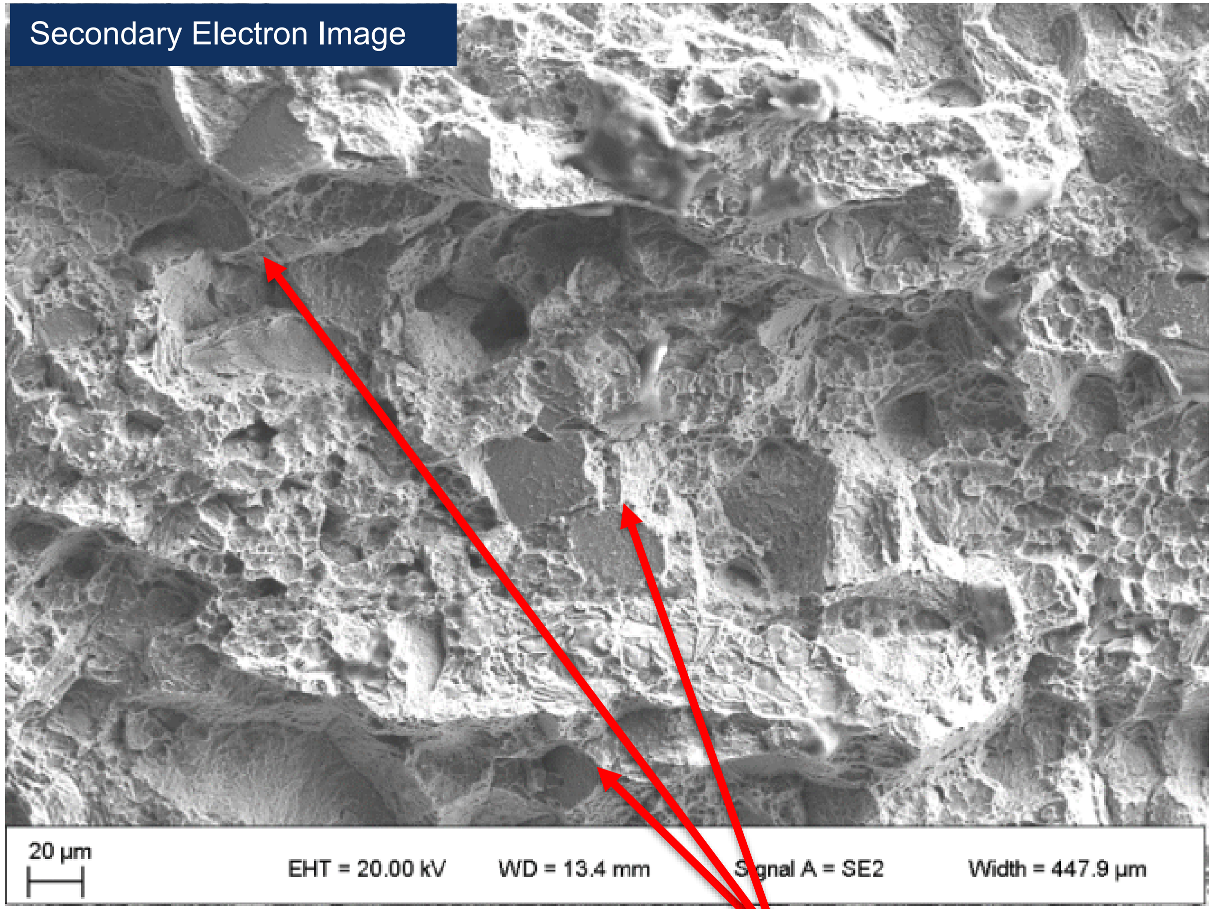
Lessons Learned

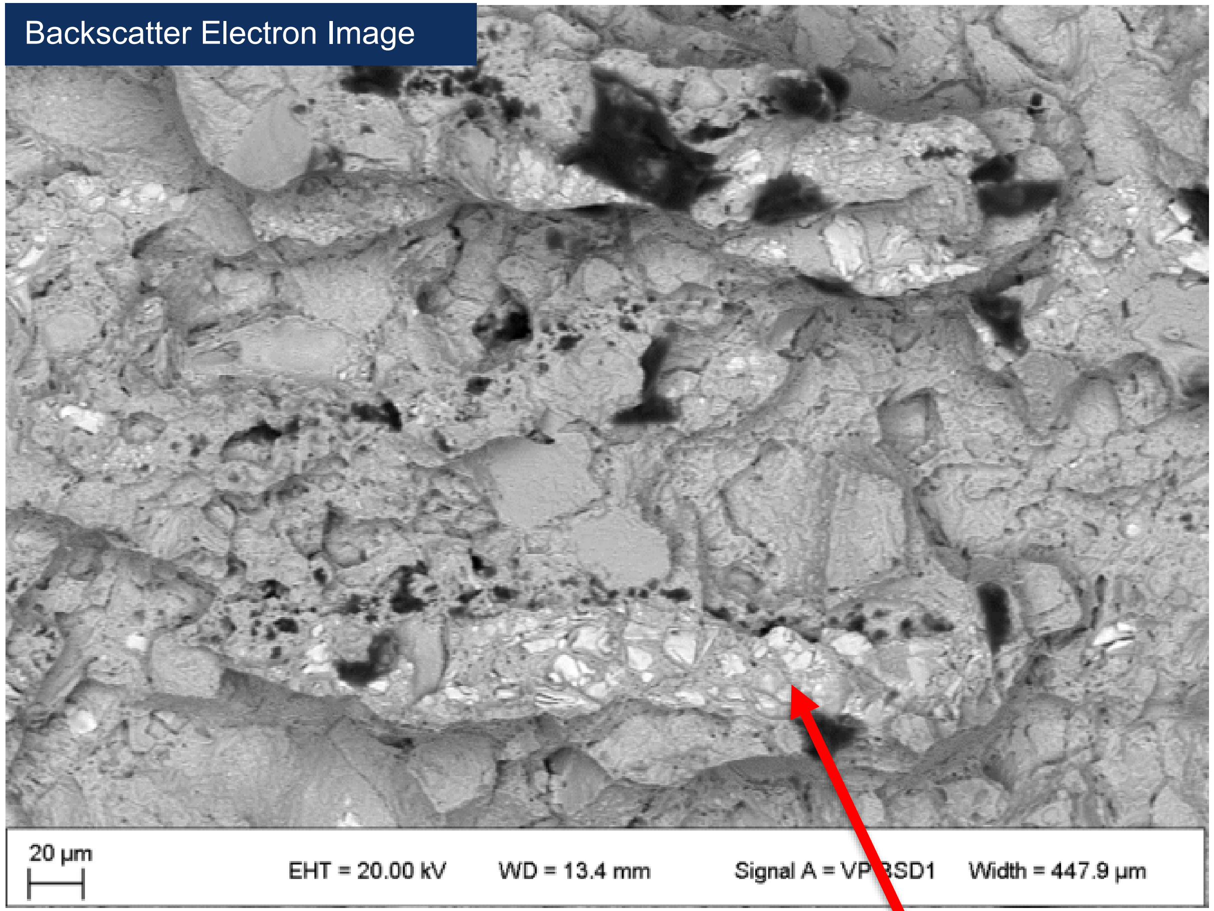
- *"They would never make it like that!":* Don't assume a vendor will make the part in a metallurgically prudent manner.
- Be aware of alternate material call-outs. Alternate material call-outs may not be equivalent.
- Product form is important. The product form (i.e., plate, bar, sheet, forging, casting) influences operative property anisotropy. Use caution extrapolating property data/behavior across product forms.
- Superfluous heat treatment of metal parts is rarely beneficial. Always consider potential metallurgical changes at elevated temperature.
- Even trusted vendors are not interchangeable
- Don't discount the importance of drawing reviews
- <u>The failure analysis of bellows assemblies made at Vendor #1 emphasizes the importance to consider equilibrium phase formation in Nitronic 60 stainless steel</u>

Backup Slides

Sample #2: Detail

- Higher magnification images of fracture surface show intergranular fracture, which is not expected for N60
- Nb-rich (bright particles in backscatter image) distributed along apparent rolling direction





SEM of intergranular second phase

- Cracking observed within Nb-rich phase (bright particles)
- Intergranular cracking along second phase was observed in addition to cracking localized near ferrite stringers oriented normal to the loading direction.

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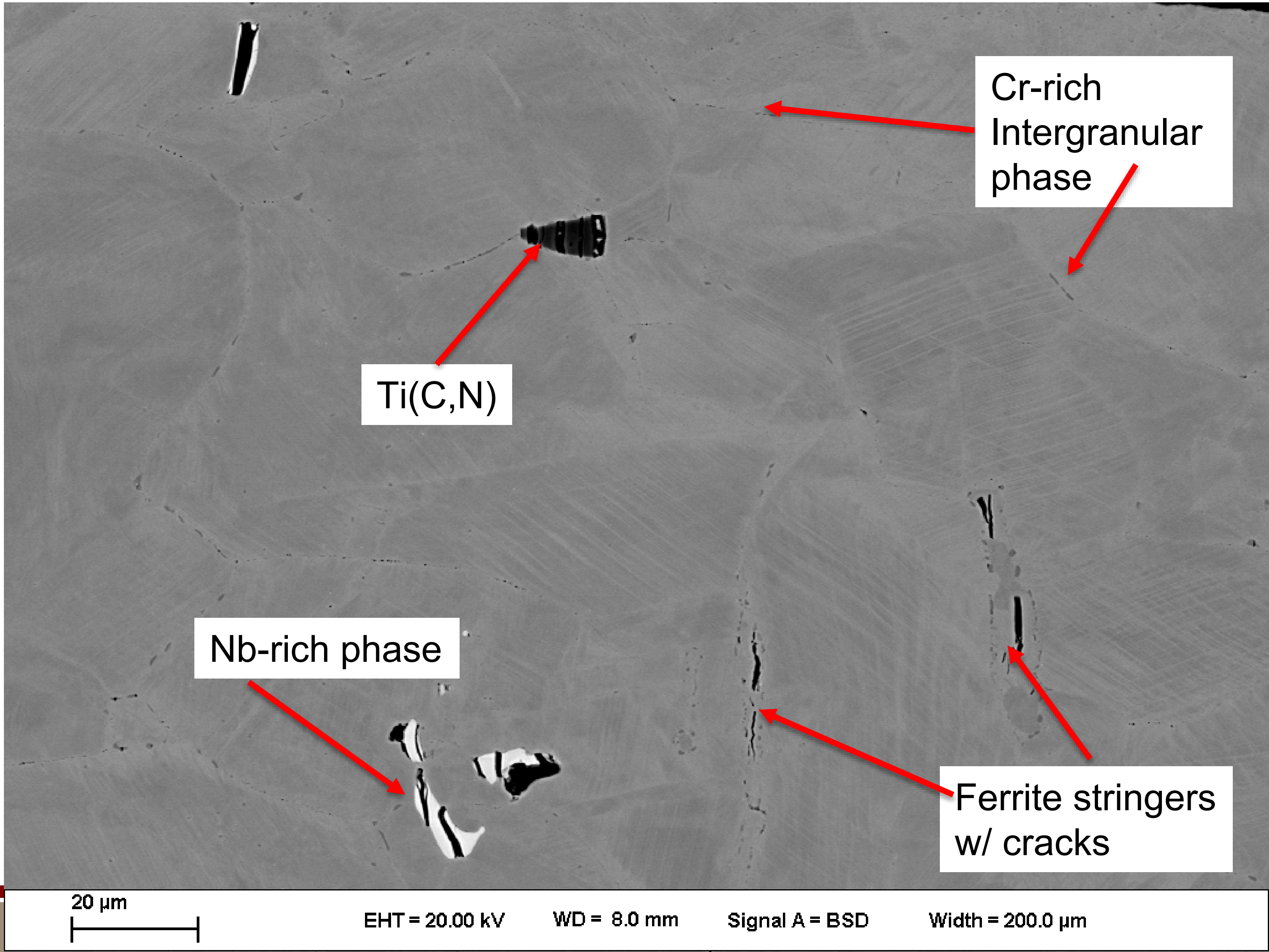
Loading Direction

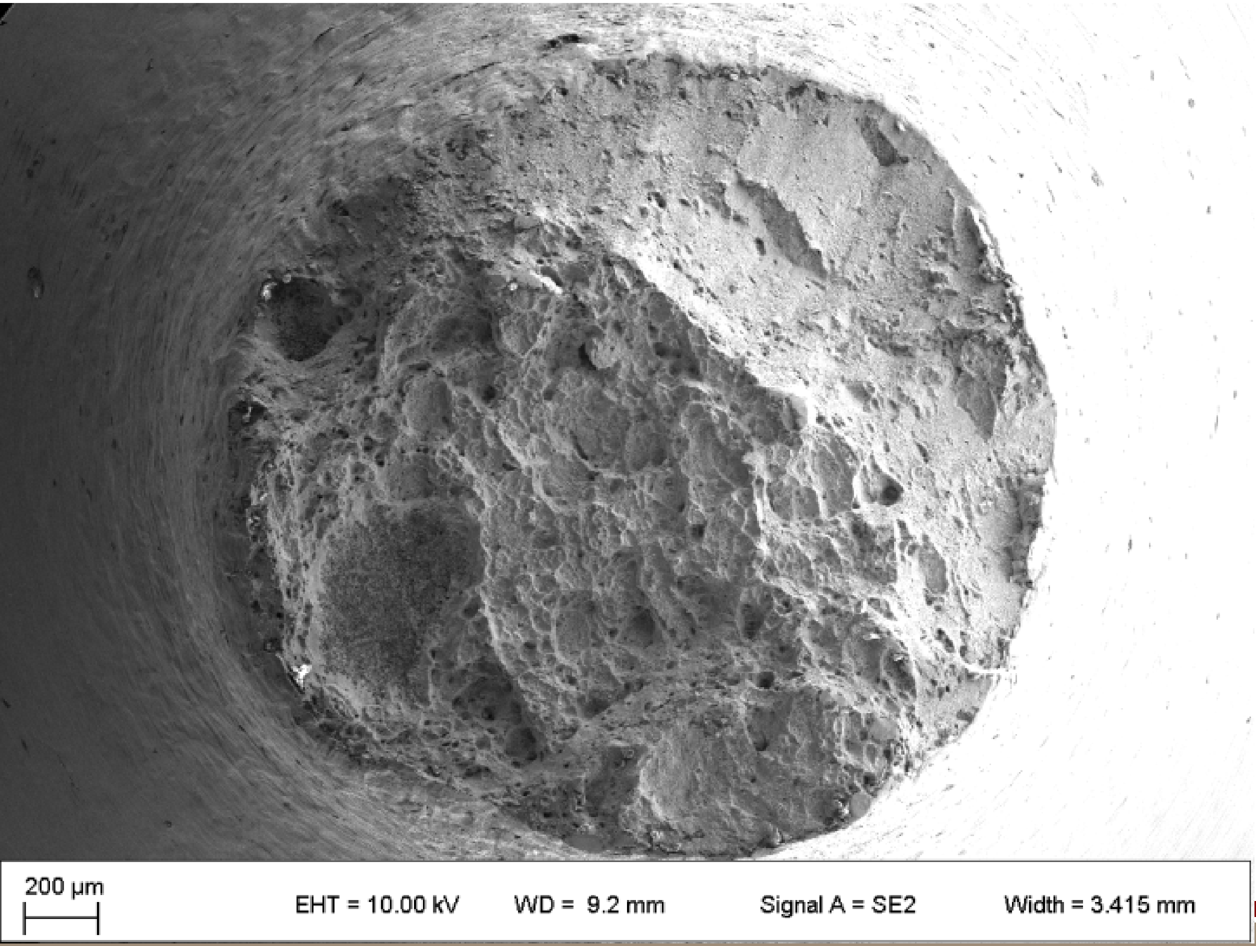
<u>Sample #6</u>



Fractography Samples #2 and #4

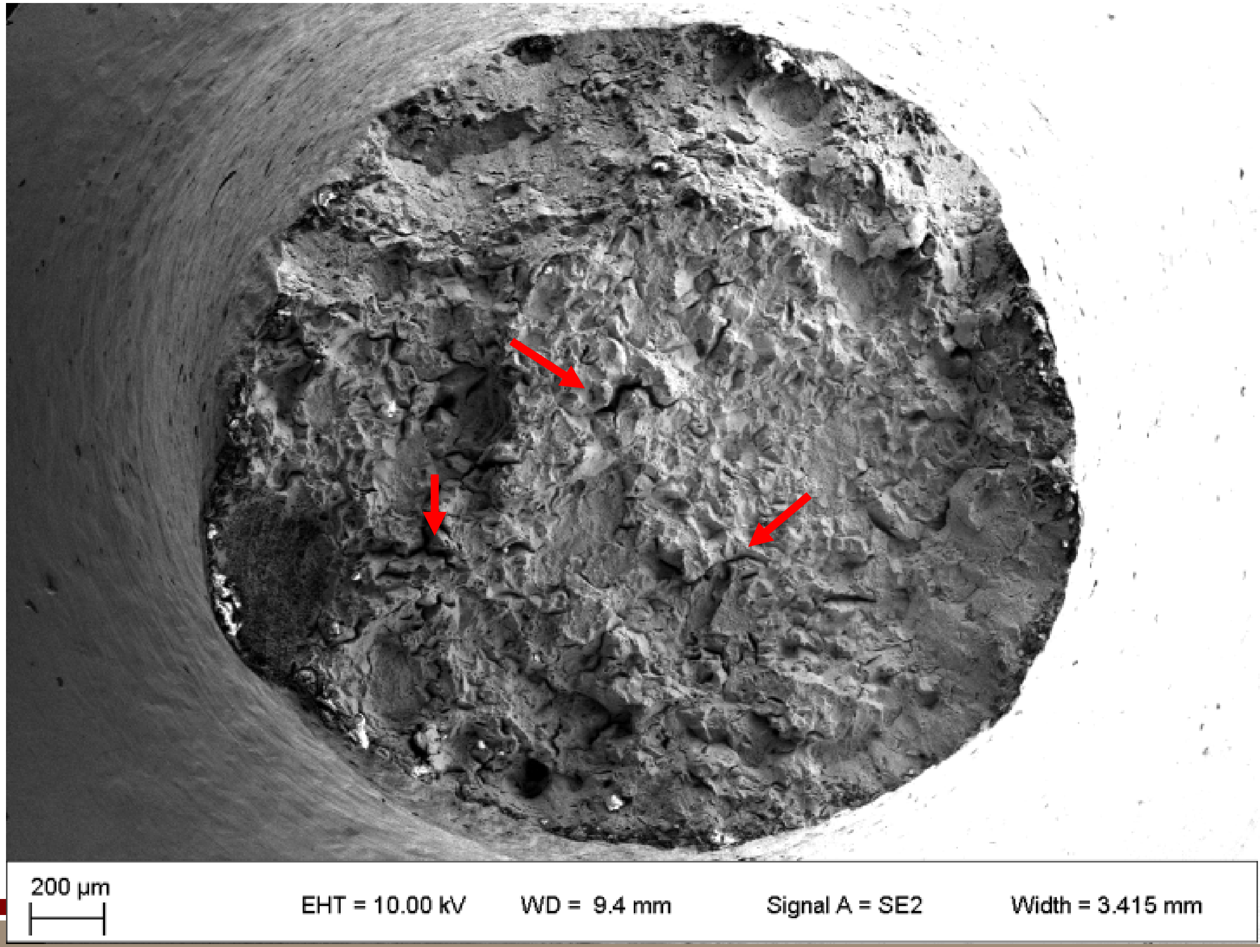
No Heat Treat – Sample #2

- Fracture surface shows typical transgranular ductile fracture expected for stainless steel at low magnification



Heat Treated – Sample #4

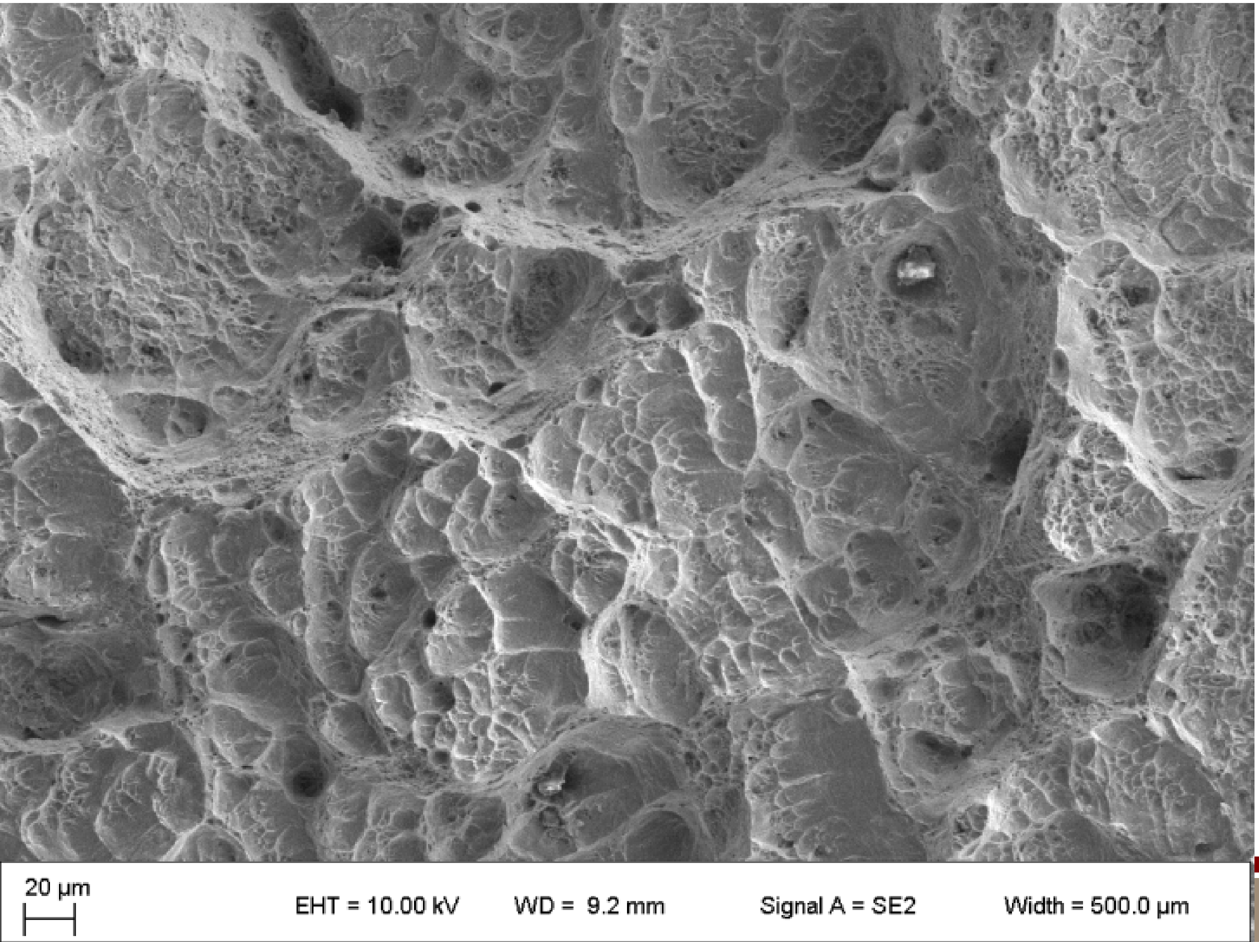
- Secondary cracking and intergranular fracture visible at low magnification. These fracture features are not expected for austenitic stainless steel.



Fractography Samples #2 and #4 (detail)

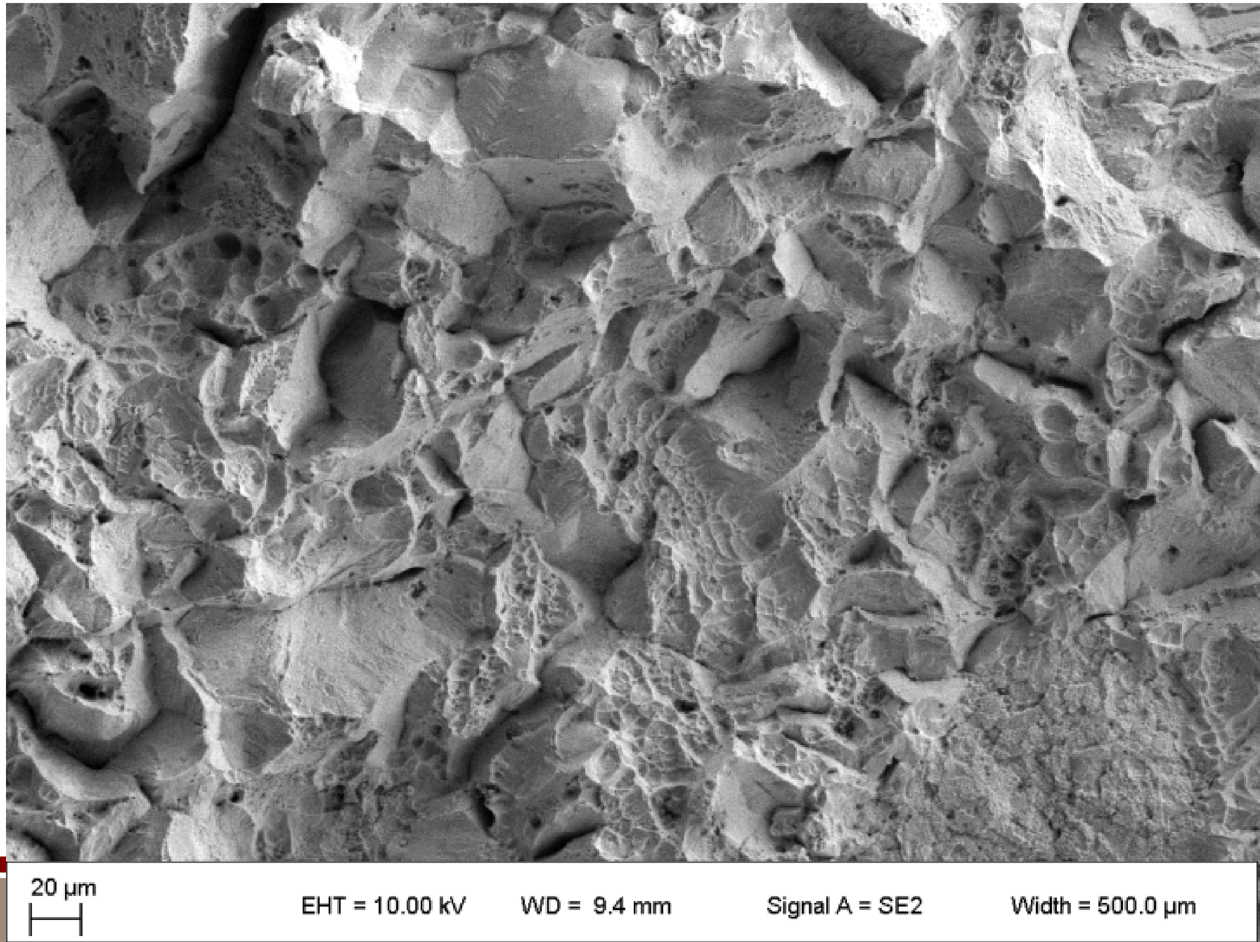
No Heat Treat – Sample #2

- Higher magnification examination of N60 tensile sample without heat treatment show expected ductile transgranular fracture behavior. No intergranular fracture observed for sample #2.



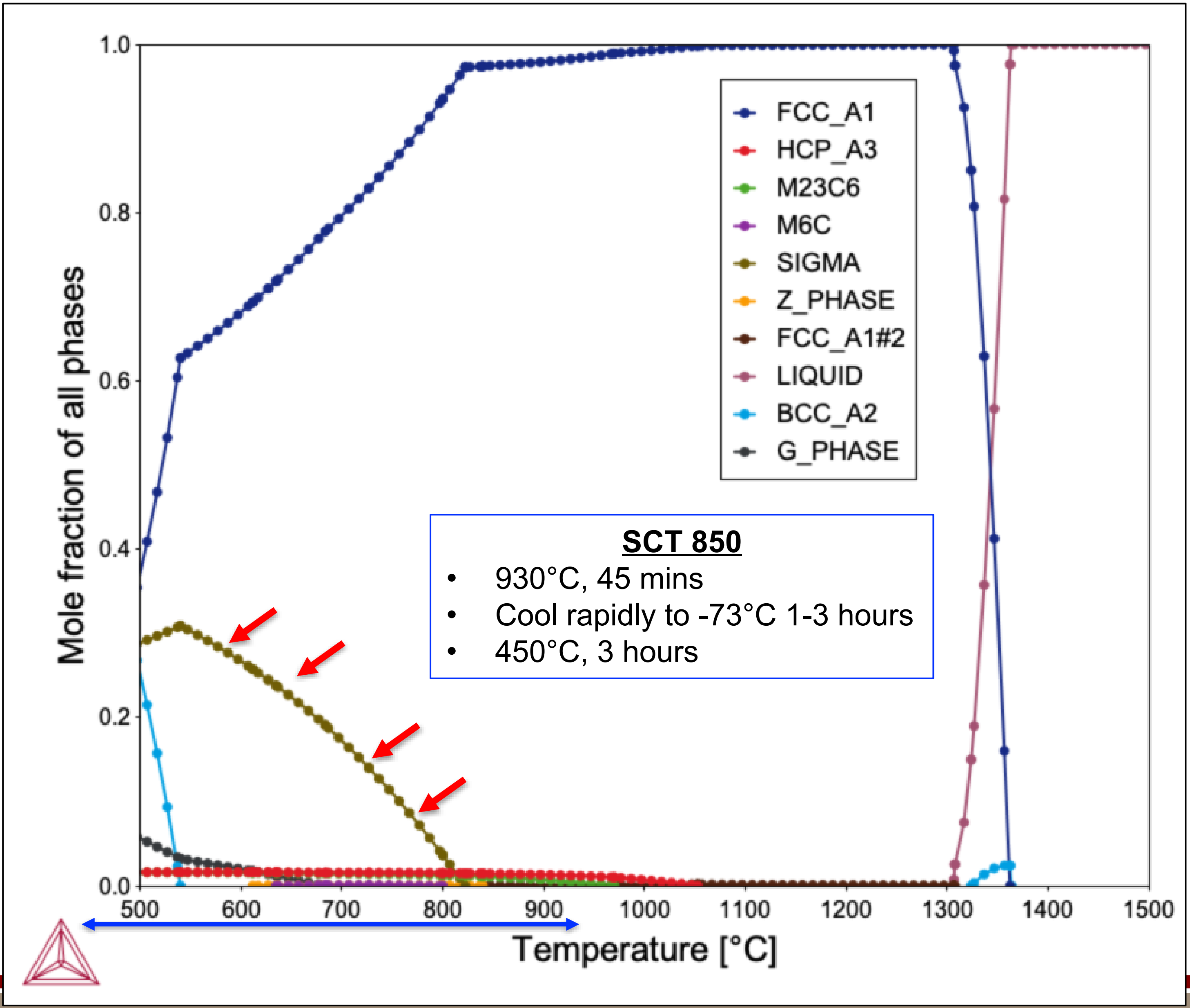
Heat Treated – Sample #4

- Mixed-mode fracture morphology observed for heat treated sample.
- Intergranular fracture (i.e., grain facets clearly visible) with secondary cracking indicative of operative embrittlement



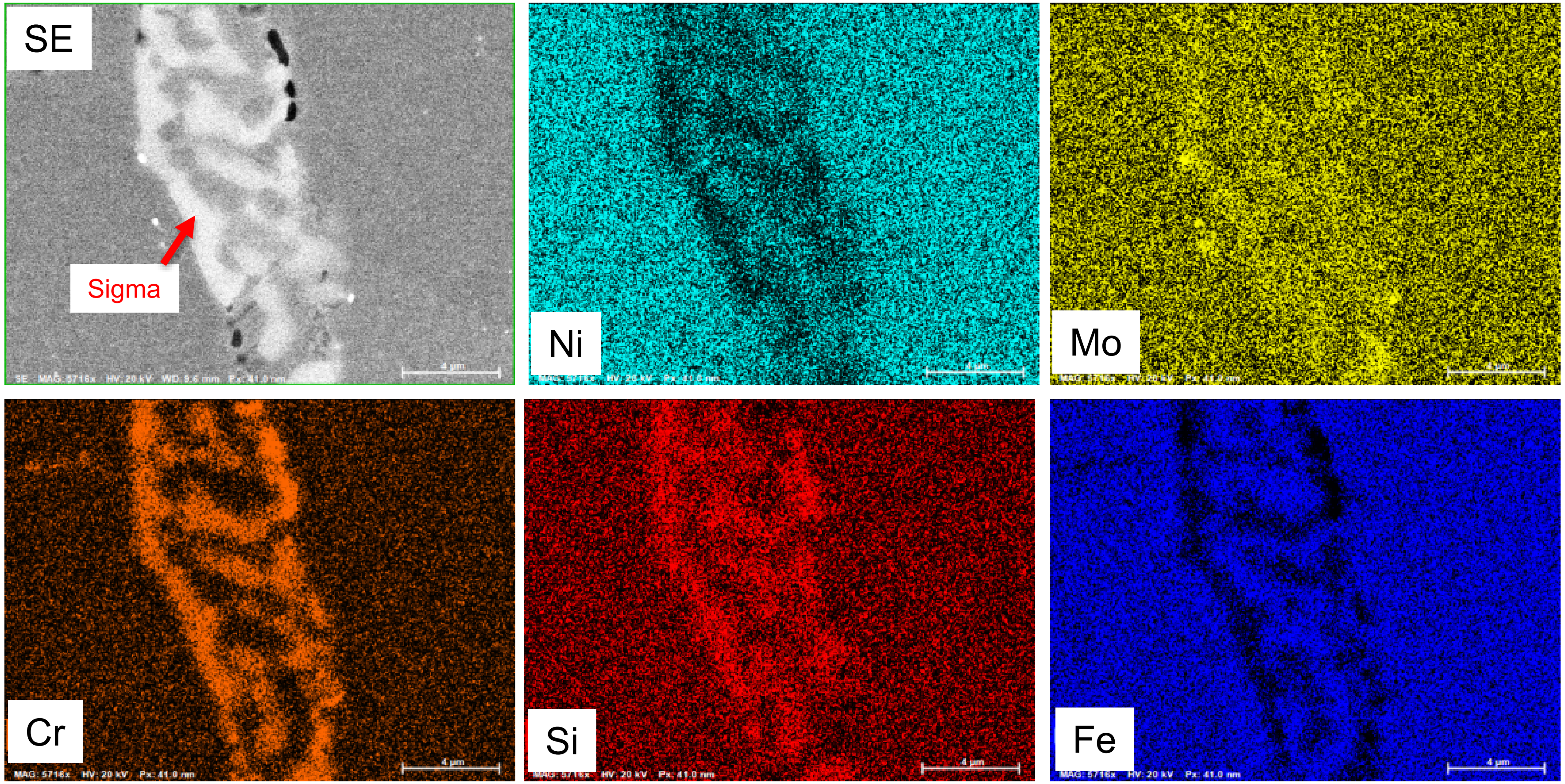
Multicomponent Thermodynamic Modeling of N60

- Multicomponent thermodynamic modeling of N60 using the CALPHAD method was performed to predict equilibrium phases in N60 for SCT 850 heat treatment conditions
- TCFE8 thermodynamic database was used to calculate temperature-dependent phase constitution for Lot 1 tensile samples using 3rd party chemistry data
- 9 component simulation performed: Fe-16.50Cr-8.38Mn-8.07Ni-3.98Si-0.075C-0.140N-0.058Nb-0.29Mo (wt.%)
- Simulation suggests that appreciable sigma phase formation for SCT 850-relevant temperatures
- Sigma is an undesirable embrittling phase in austenitic stainless steels



Sigma phase in N60 Lot 1 samples is Cr- and Si-rich consistent with thermodynamic predictions

- X-ray EDS measurements of sigma phase located in preexisting ferrite stringers is in good agreement with thermodynamic predictions. Sigma for N60 is Si-rich.



<u>Multicomponent thermodynamic prediction of sigma phase composition for Lot 1 samples</u>