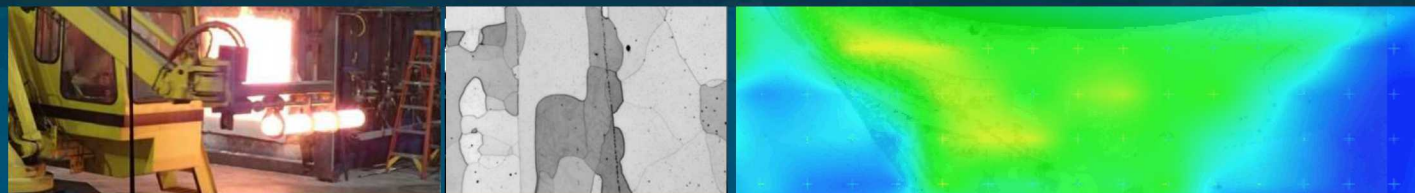


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SAND2019-6885C

Low-Sulfur Magnetic Stainless (LSMS): An Overview



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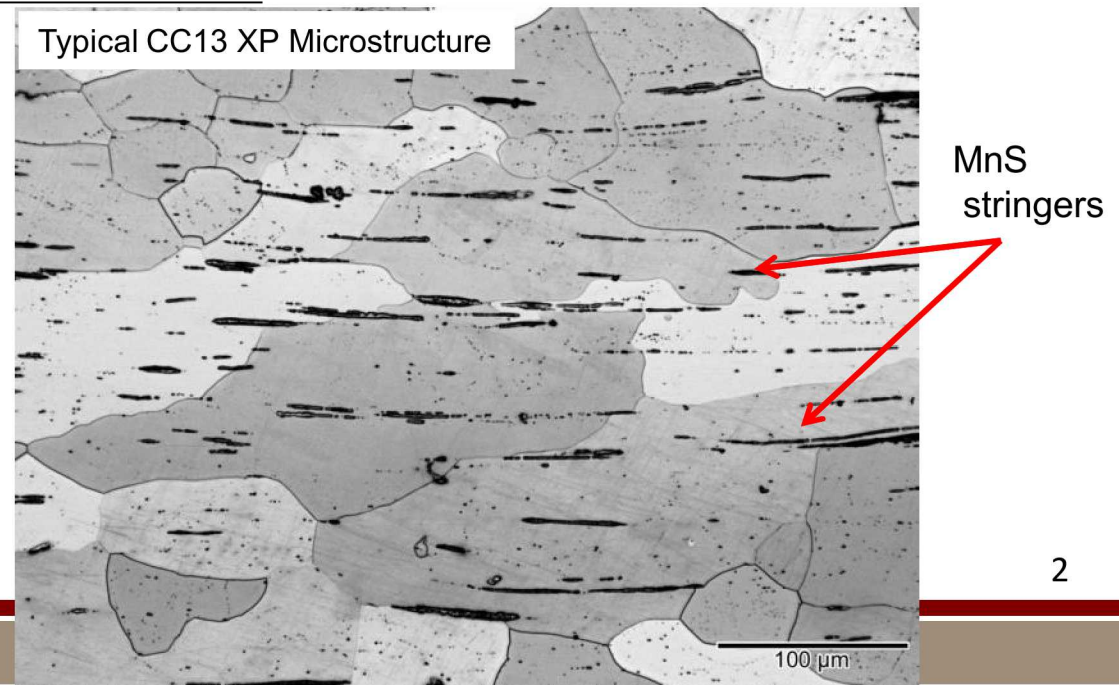
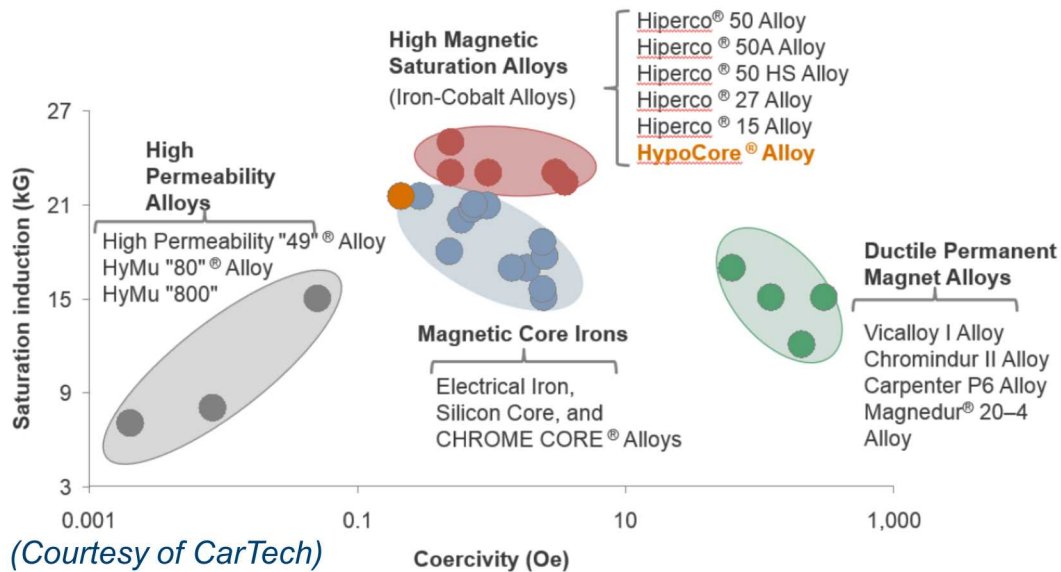
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Ferritic stainless steels are promising option for non-hermetic compact actuation applications

- Stainless soft magnetic alloys based on the ferritic stainless steel systems offer intrinsic corrosion resistance
- ChromeCore® 13XP (CC13XP), a commercially-produced 13Cr ferritic stainless steel manufactured by Carpenter Technology, was identified as candidate soft magnetic alloy for non-hermetic solenoid applications
- CC13XP is regarded as a 'free-machining' alloy due to the high-concentration (~2000—3000 wt.ppm) of sulfur added to form MnS stringers
- Presence of MnS stringers compromises corrosion resistance in aggressive environments

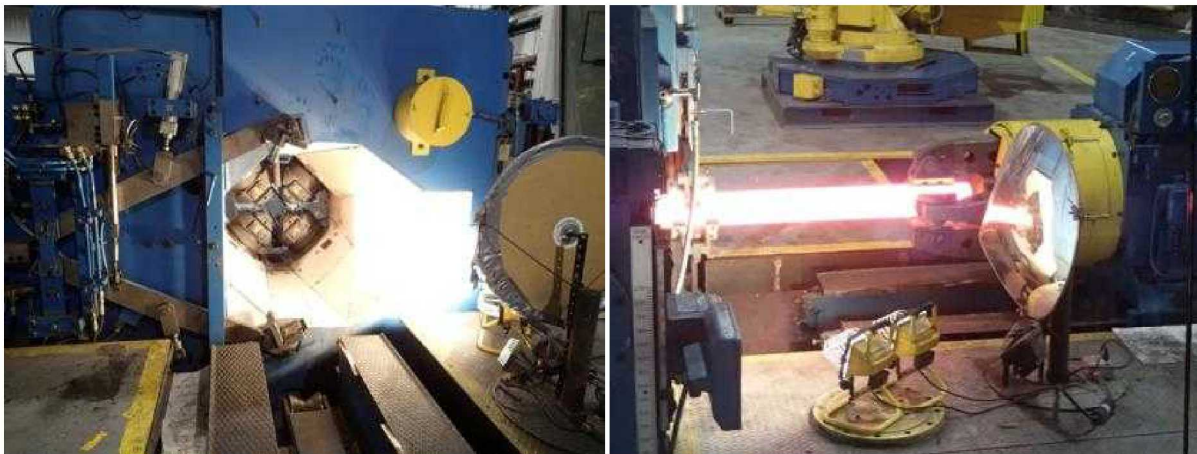


SNL-NM teamed with specialty alloy producer to produce development heat of Low-S CC13XP (renamed: Low-S Magnetic Stainless [LSMS])

- Sulfur content was reduced ~100X to eliminate MnS network and improve corrosion resistance
- An evaluation-sized (500#) ingot of vacuum induction melted LSMS was cast and forged by specialty alloy producer
- Gyrotrary forging (GFM) was used to reduce 6" dia. cast ingot to ~1.2" dia. round bar
 - Bar was finished to 1.00" dia.
- Resulting yield:
 - 116 lbs. (6 ea. ~90" lengths) heat treated 850°C for 3.5 hrs. (**magnetic anneal**)
 - 120 lbs. (6ea. ~90" lengths) heat treated 650°C for 1 hr. (mill anneal)

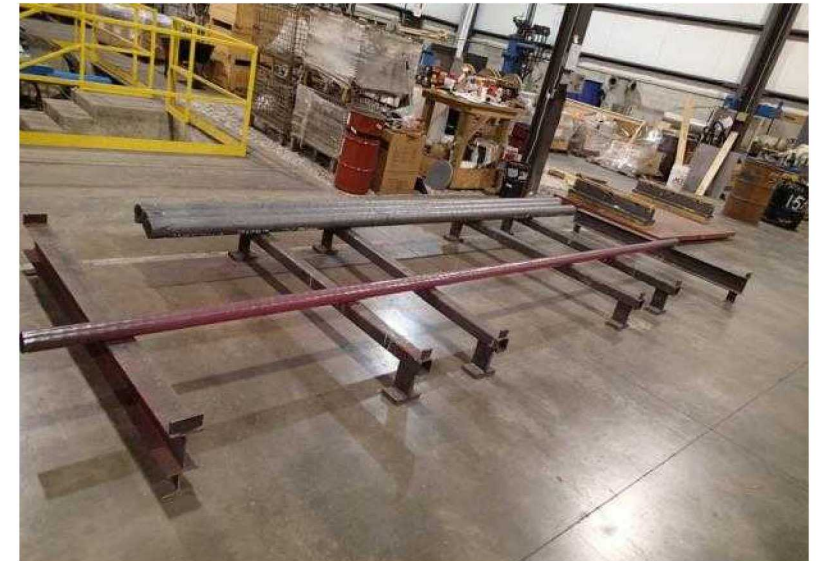


6" diameter ingot is heated to 1150°C in a furnace and is immediately transferred to the GFM



Left: GFM head with 4 reciprocating hammers. Hammers oscillate approximately 500 times/min. with each hammer strike delivering ~250 tons of forging force

Right: Ingot is continuously rotated and fed into GFM hammers

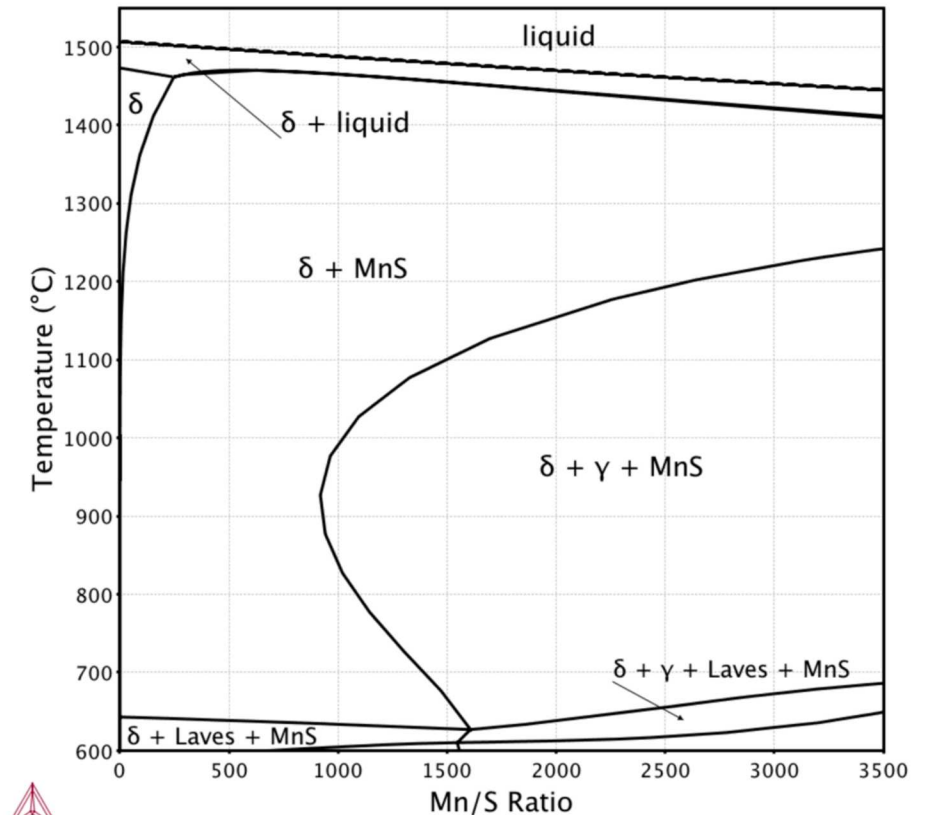


Ingot is reduced to 3" dia. bar and allowed to cool to room temperature. ~20' length will be sectioned in 3' lengths, reheated, and forged down to ~1.2" dia.

Resultant Alloy Composition of LSMS Lot #1

- Nearly 100X reduction in sulfur content for LSMS compared to CC13XP
- Principal element concentrations for LSMS mimic CC13XP—except for Mn
- Multicomponent thermodynamic simulations used to understand effect of ‘free’ Mn

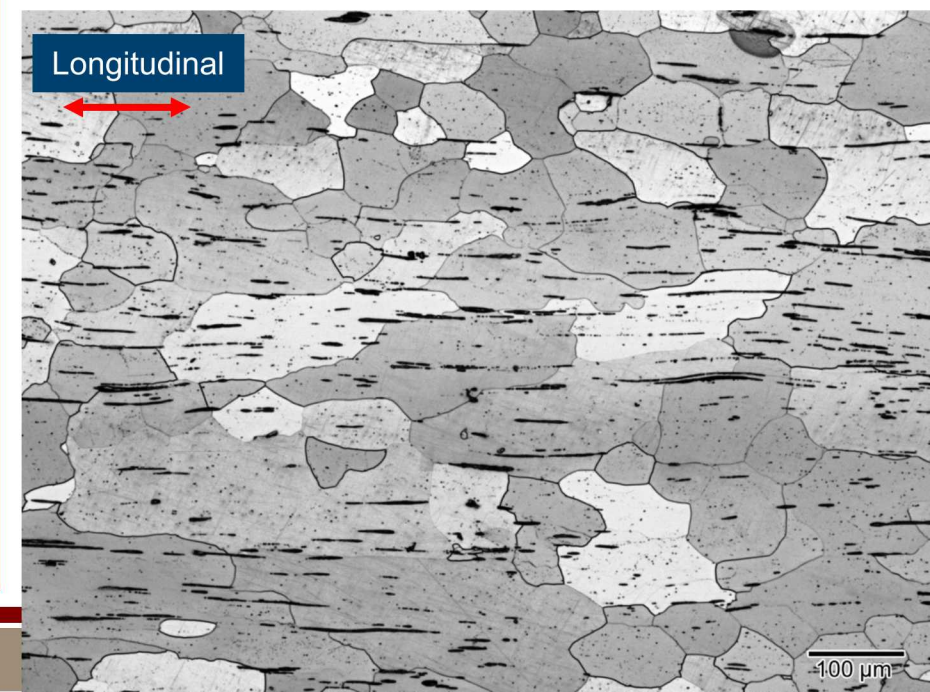
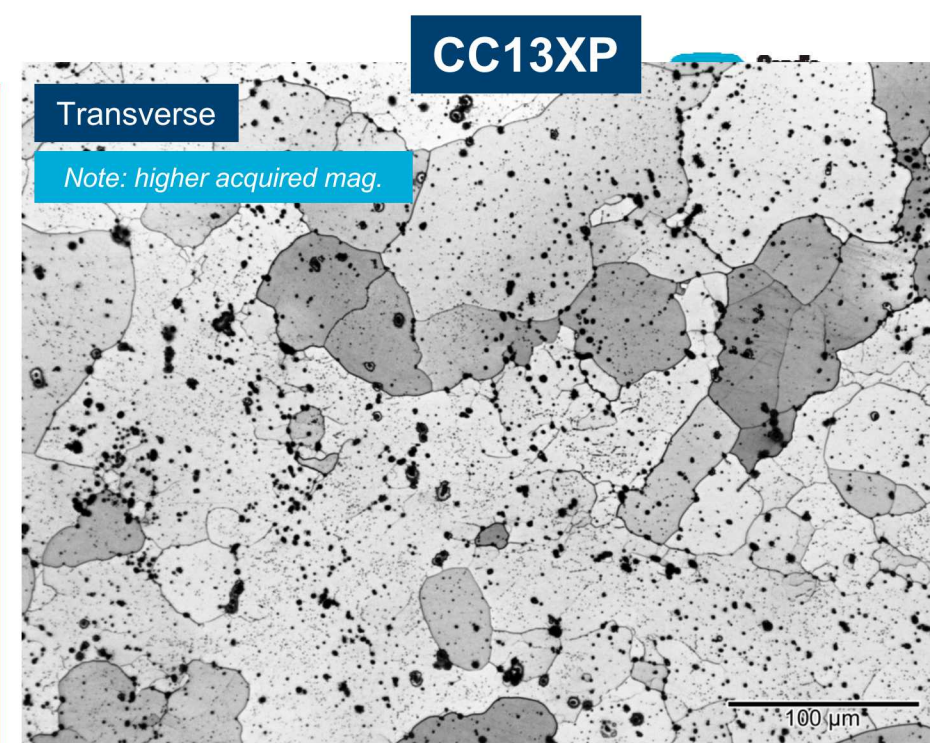
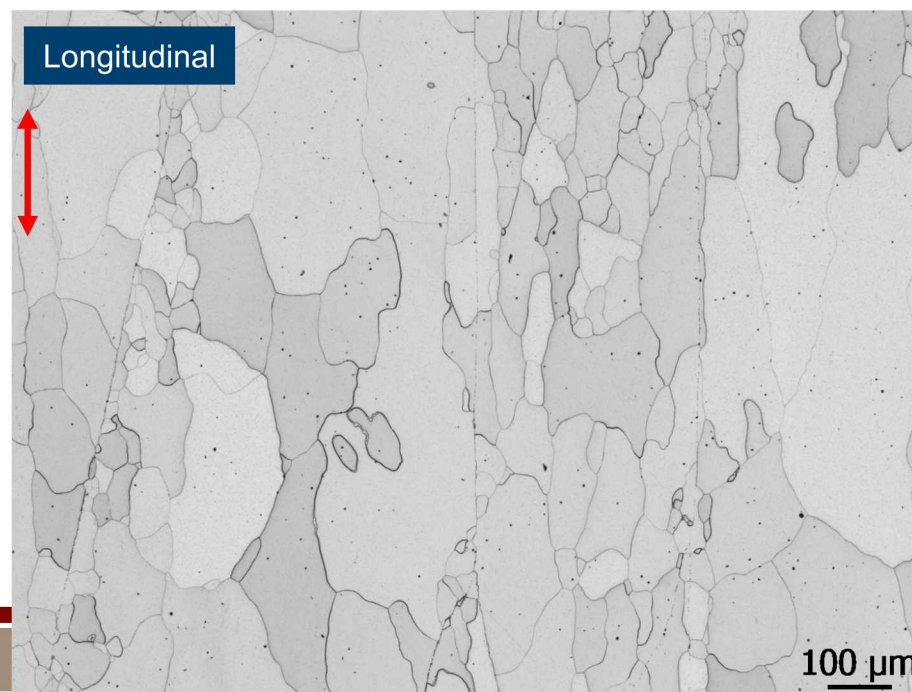
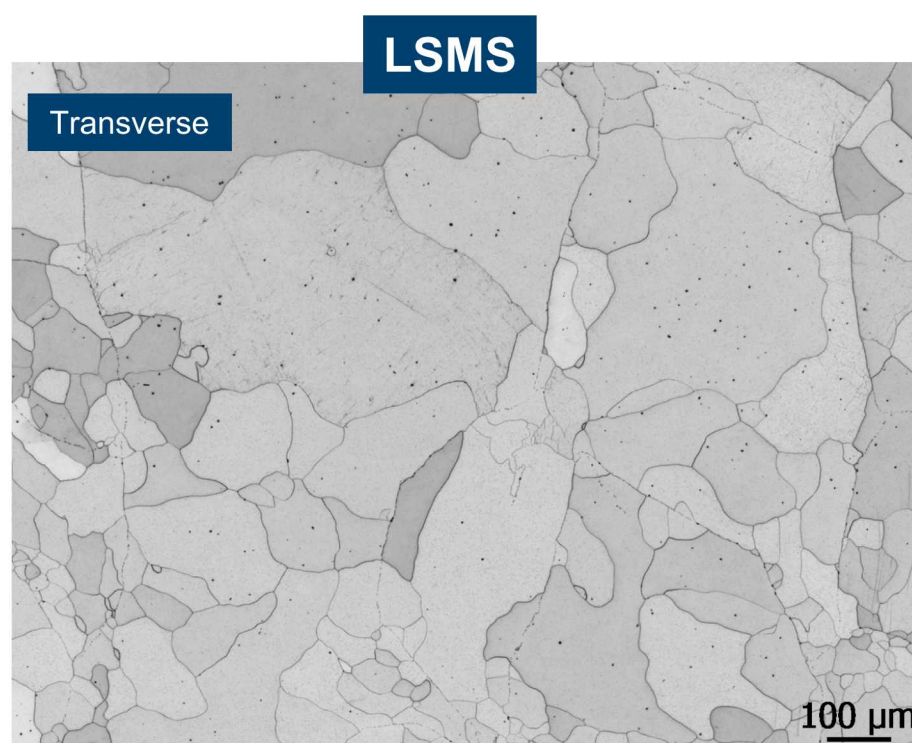
Element	Target LSMS Composition Range (wt%)	LSMS: Lot #1: M723260 (wt%)	Carpenter CC13XP heat: 734539 (wt%)
C	0.03 max.	0.009	0.013
P	0.03 max.	<0.005	0.016
Si	1.20-1.80	1.27	1.46
Mo	0.50-1.00	0.78	0.76
Mn	0.20 max.	0.006	0.42
Cr	12.50-13.50	13.04	12.94
V	0.5-1.00	0.75	0.74
S	0.005 max.	0.003	0.28
N	report only	0.004	0.011
Ni	report only	0.01	0.3
Cu	report only	<0.01	0.04
Co	report only	0.01	0.01
Al	report only	0.01	0.003
Fe	balance	balance	balance



Effect of Mn/S ratio on phase stability (avoid austenite)

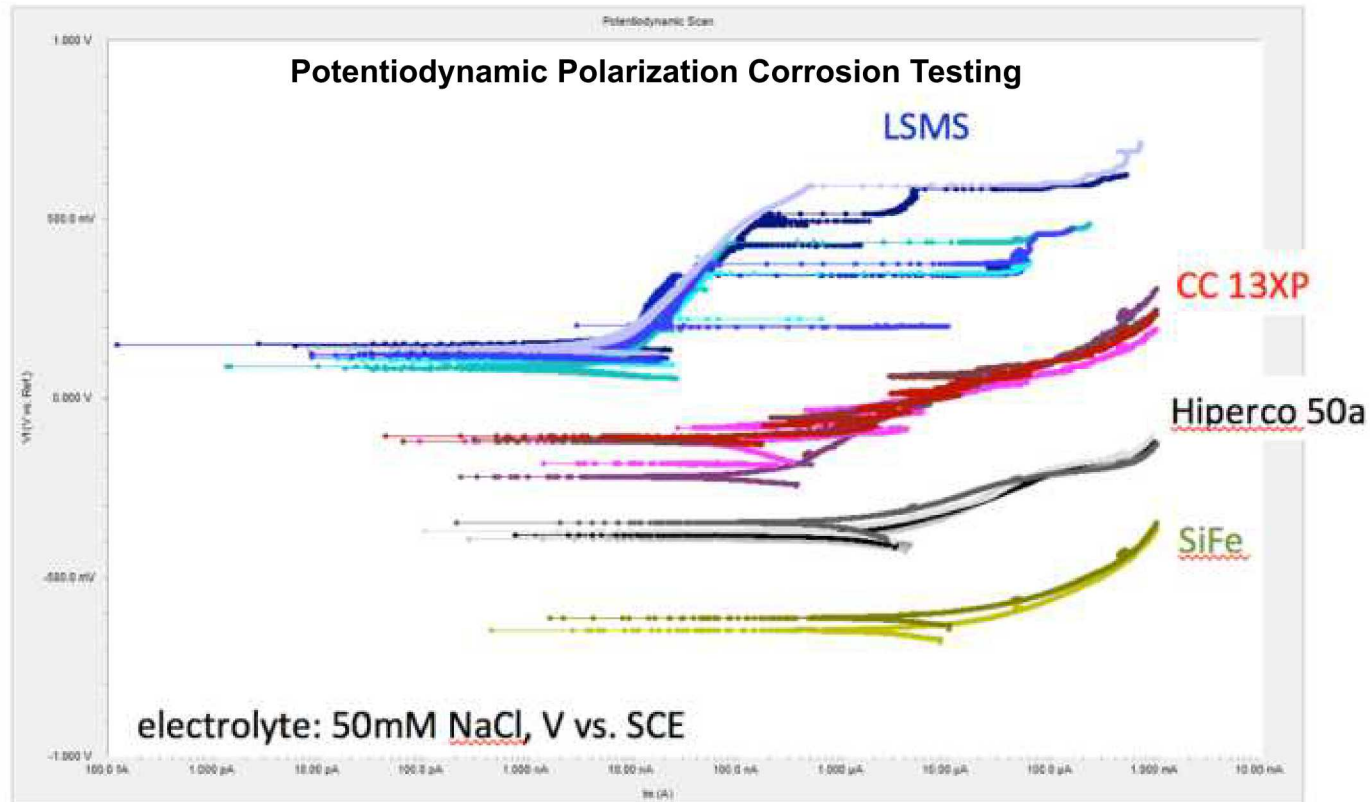
LSMS vs. CC13XP: General microstructure

- Both CC13XP and LSMS in magnetic annealed condition show equiaxed ferrite grains
- LSMS is essentially devoid of second-phase compared to CC13XP which contains semi-continuous network of MnS stringers

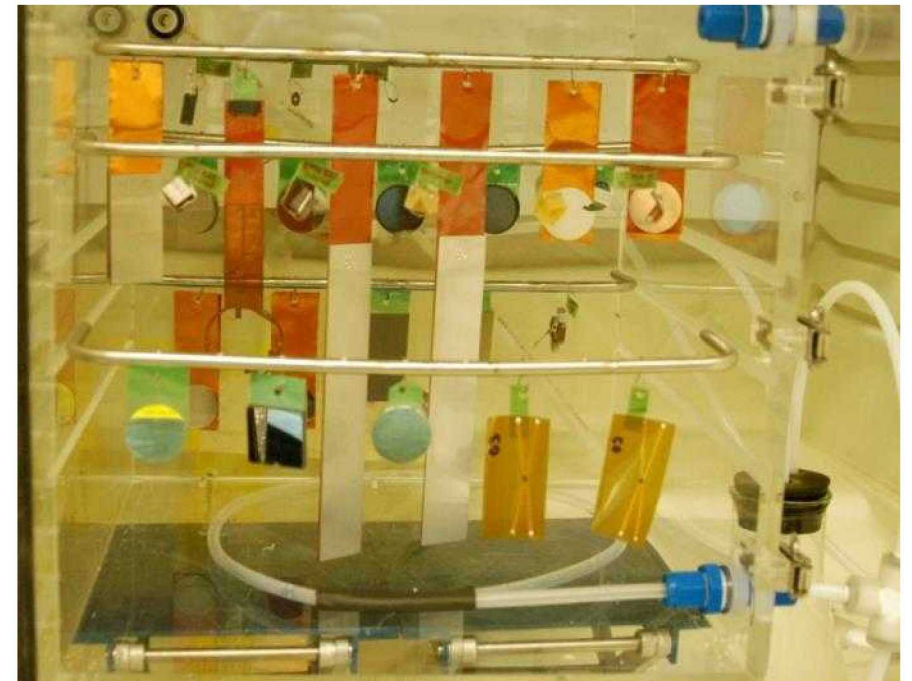


Reduction in sulfur content for LSMS improves corrosion resistance

- Electrochemical testing of LSMS and comparable alloys shows improved corrosion resistance via increased stability of passive region and lower open circuit current density
- CC13XP shows limited passivity and metastable pit formation



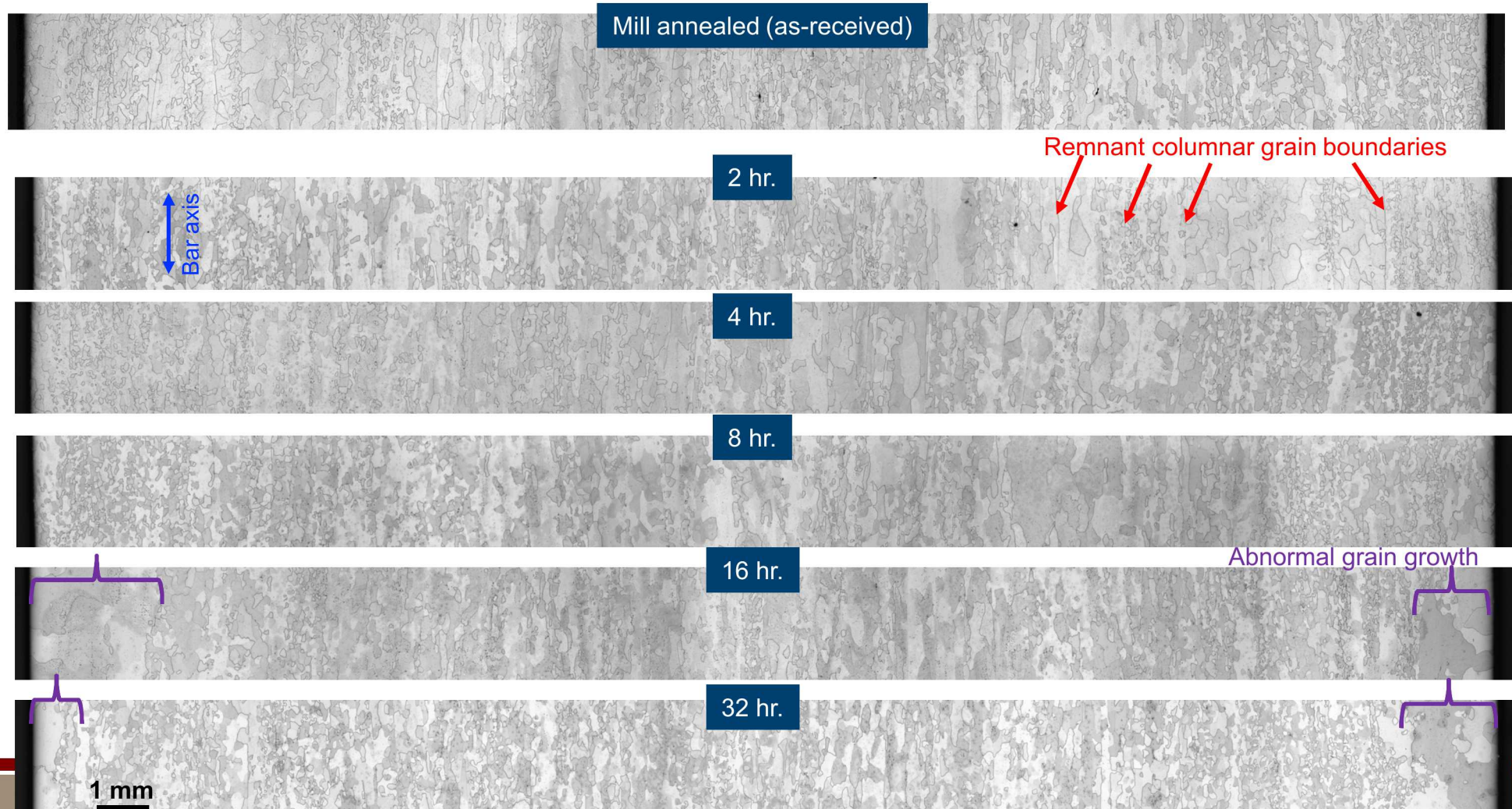
Gaseous Exposure Testing for Material Compatibility



CC13XP suffered discoloration/pitting

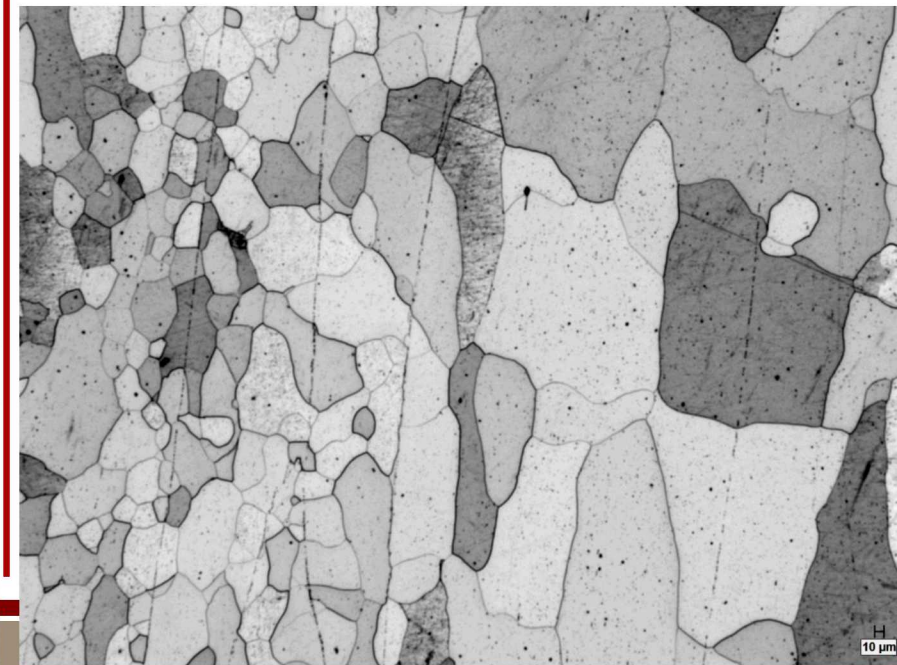
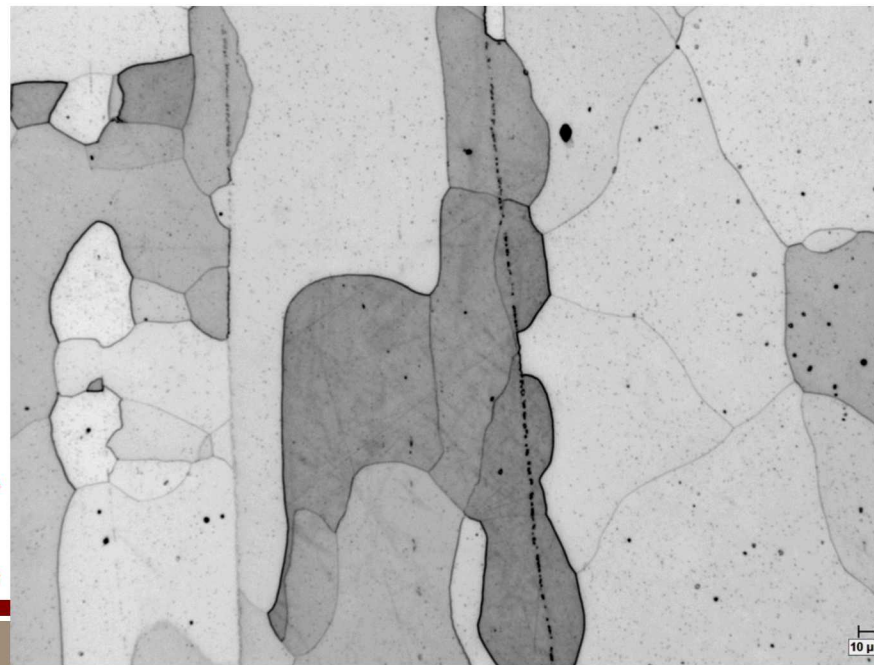
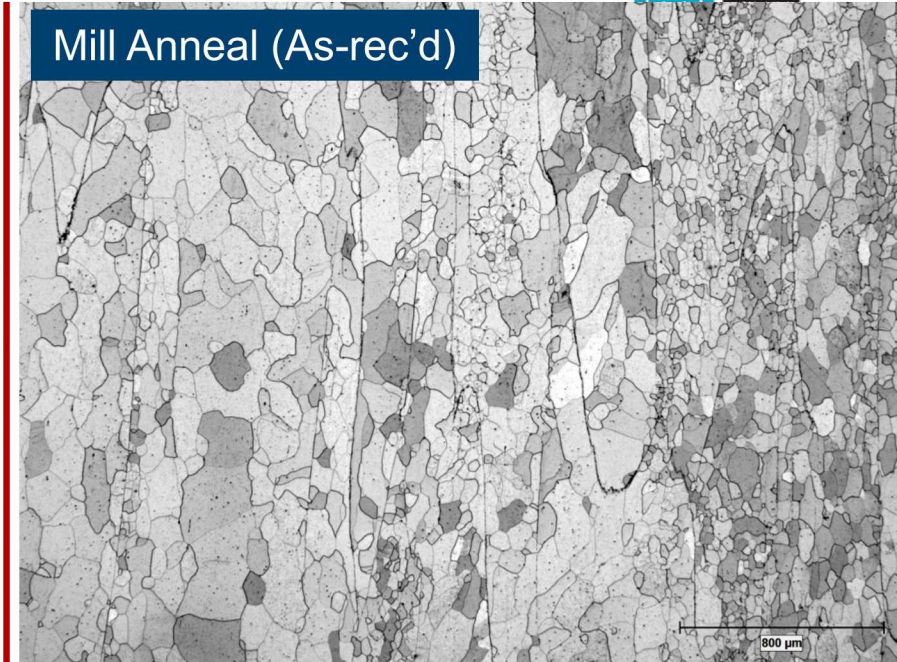
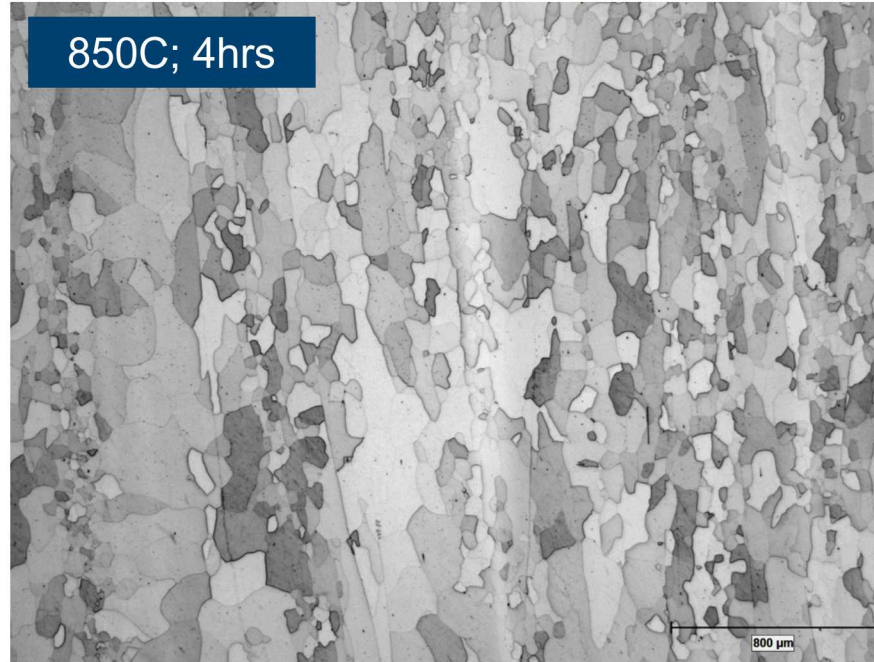
Microstructure Post-Heat Treatment (850C)

- LSMS is annealed nominally at 850C to ideally produce strain-free, recrystallized grains
- Abnormal grain growth observed on bar OD for 16 hrs. or longer soak duration



Detail: Microstructure Post Anneal

- 850C for 4 hours is nominal magnetic annealing heat treatment for both LSMS and CC13XP
- Remnant columnar grain boundaries persist after heat treatment



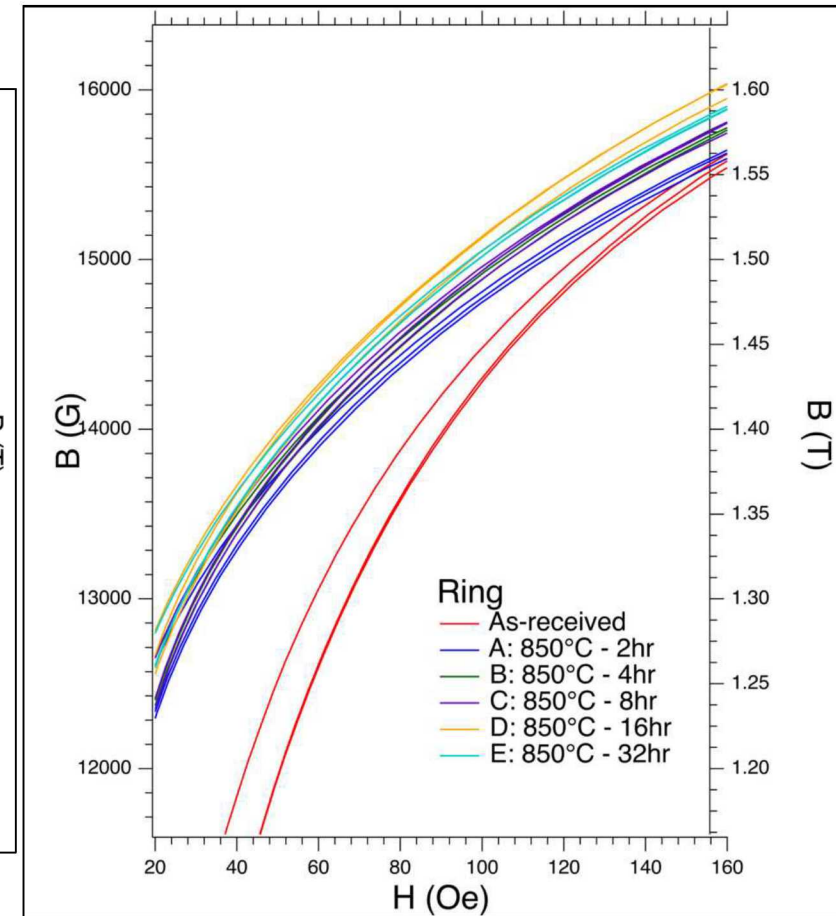
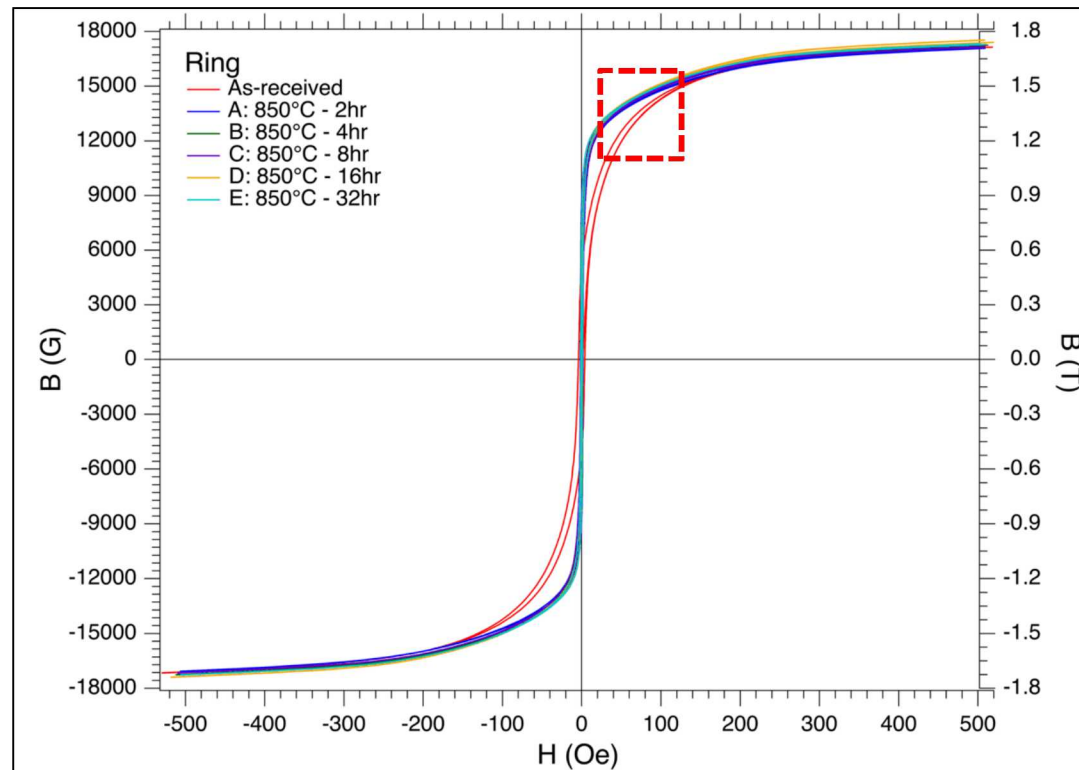
Longitudinal Section

(LSMS Lot #1; M723260)

B-H Characteristics of 850° C Heat Treated

Samples: **Ring** Geometry

- For lot of material examined, data indicates that B-H behavior for LSMS appears to be largely independent of heat treat soak time at 850° C
- At low applied field, magnetization appears to vary slightly with heat treat time. This may be attributed to change in OD bar microstructure.



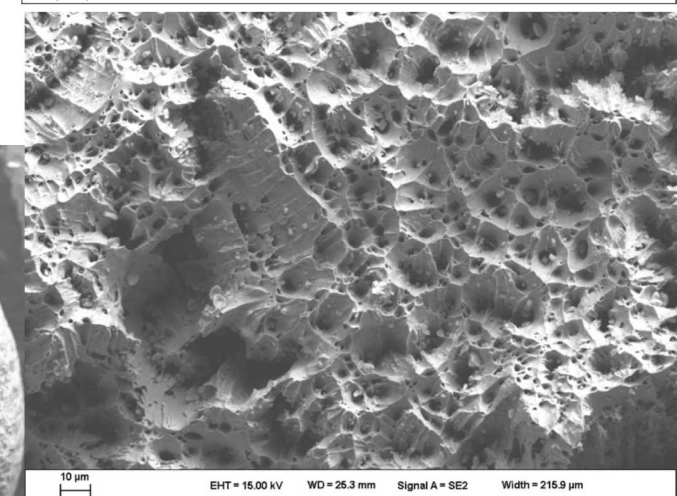
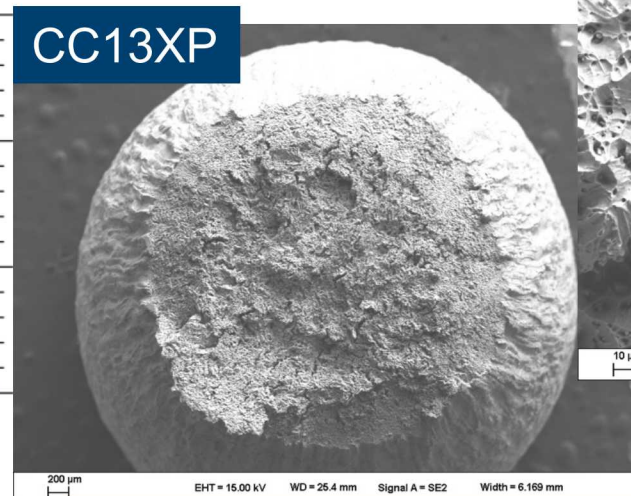
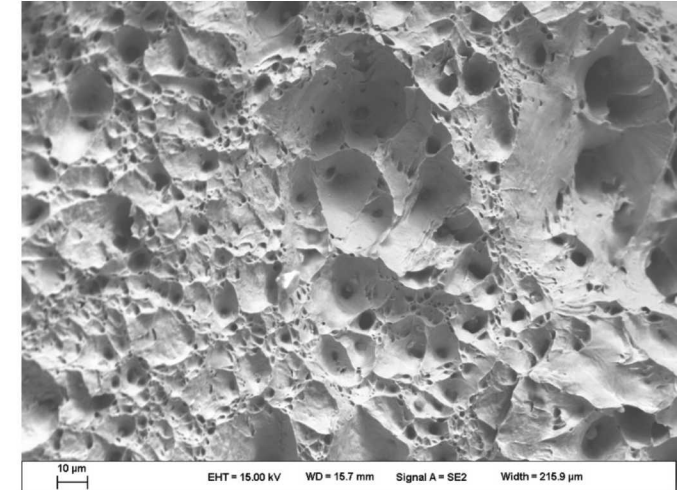
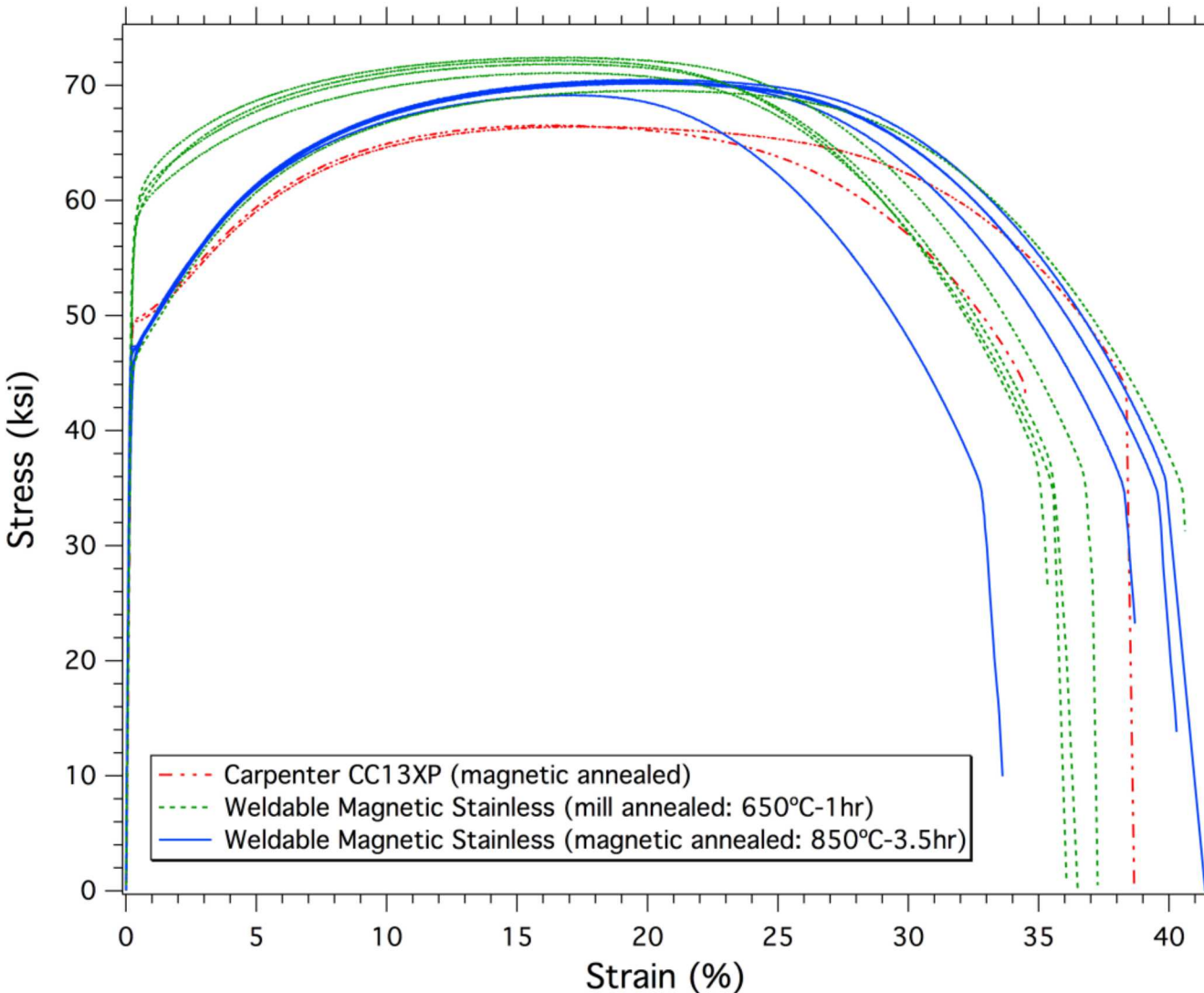
Effect of H_c on heat treatment time/temperature



Condition ID	Soak Temperature	Isothermal Hold	Ring H_c (Oe)	Bar H_c (Oe)
A	850°C	2	1.55	1.32
B	850°C	4	1.41	1.37
C	850°C	8	1.29	1.53
D	850°C	16	0.79	1.61
E	850°C	32	1.03	1.38
F	950°C	2	1.17	1.94
G	1050°C	2	1.69	1.61
H	No Heat Treat - As Received		3.96	2.74

Full Stress-Strain Curves: LSMS vs. CC13XP

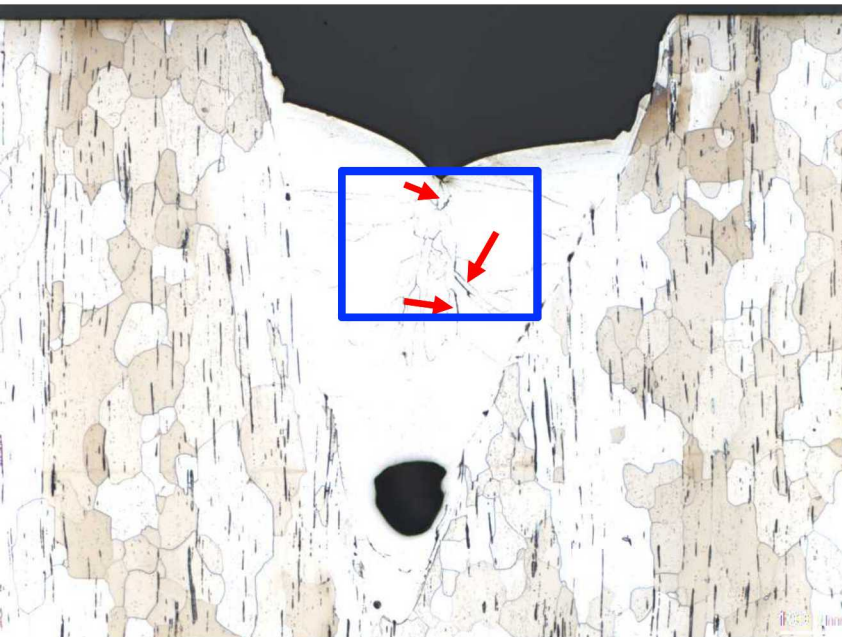
- Magnetic annealed LSMS shows similar quasi-static tensile behavior compared to CC13XP
- Unlike ordered FeCo alloys, fracture of LSMS/CC13XP occurs via necking and ductile rupture



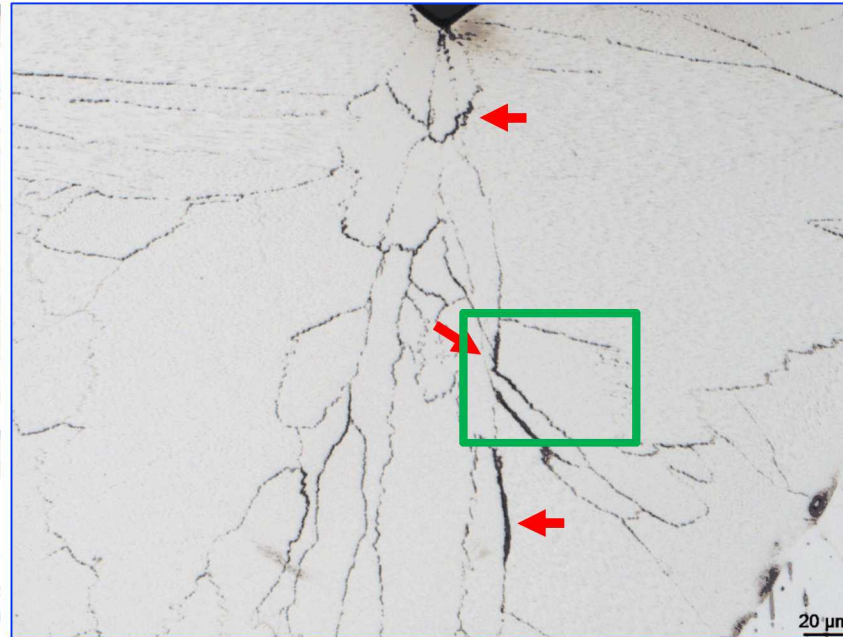
Sulfur content of CC13XP precludes welded component designs due to weld crack risk.

- Impurity elements in stainless steels (e.g., **S** and P) increase risk of cracking during weld solidification
- High S concentration (2000-3000 wt.ppm) in CC13XP results cracks when laser welded

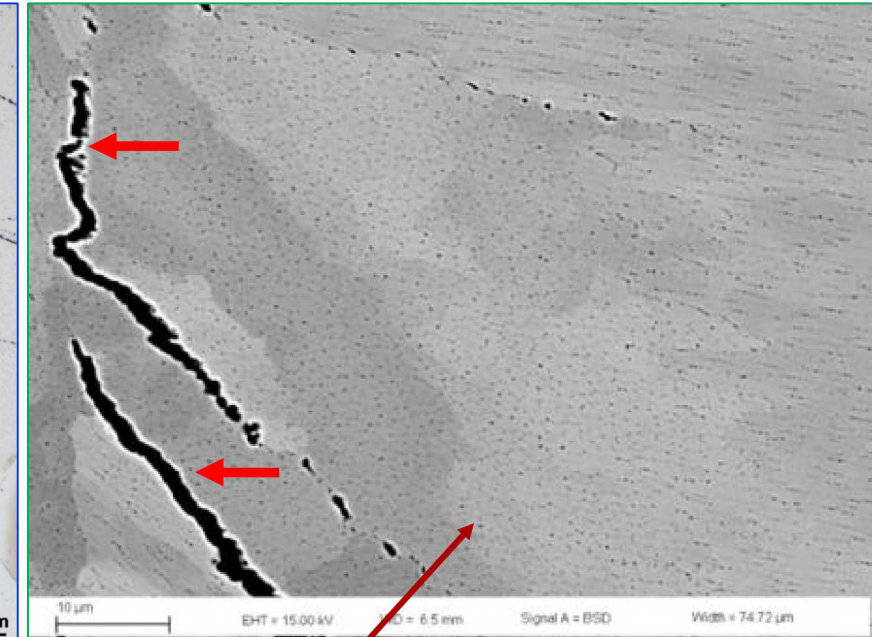
Optical Micrograph



(Autogenous pulsed laser laser)



SEM Backscatter Electron Micrograph



Dark sub-micron particles are Mn,S-rich terminal solidification phase

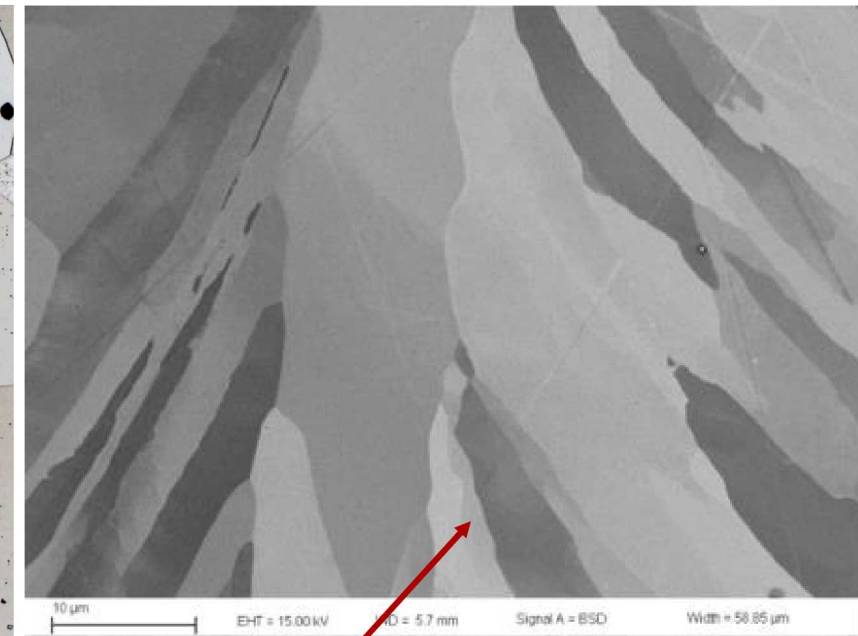
Solidification crack-free welds can be produced on LSMS due to reduction in sulfur content

- Low impurity content of LSMS (P+S <40 wt.ppm, typical) is expected to carry low risk of solidification cracking based on SNL trials and established weldability data for stainless steels

Optical Micrograph



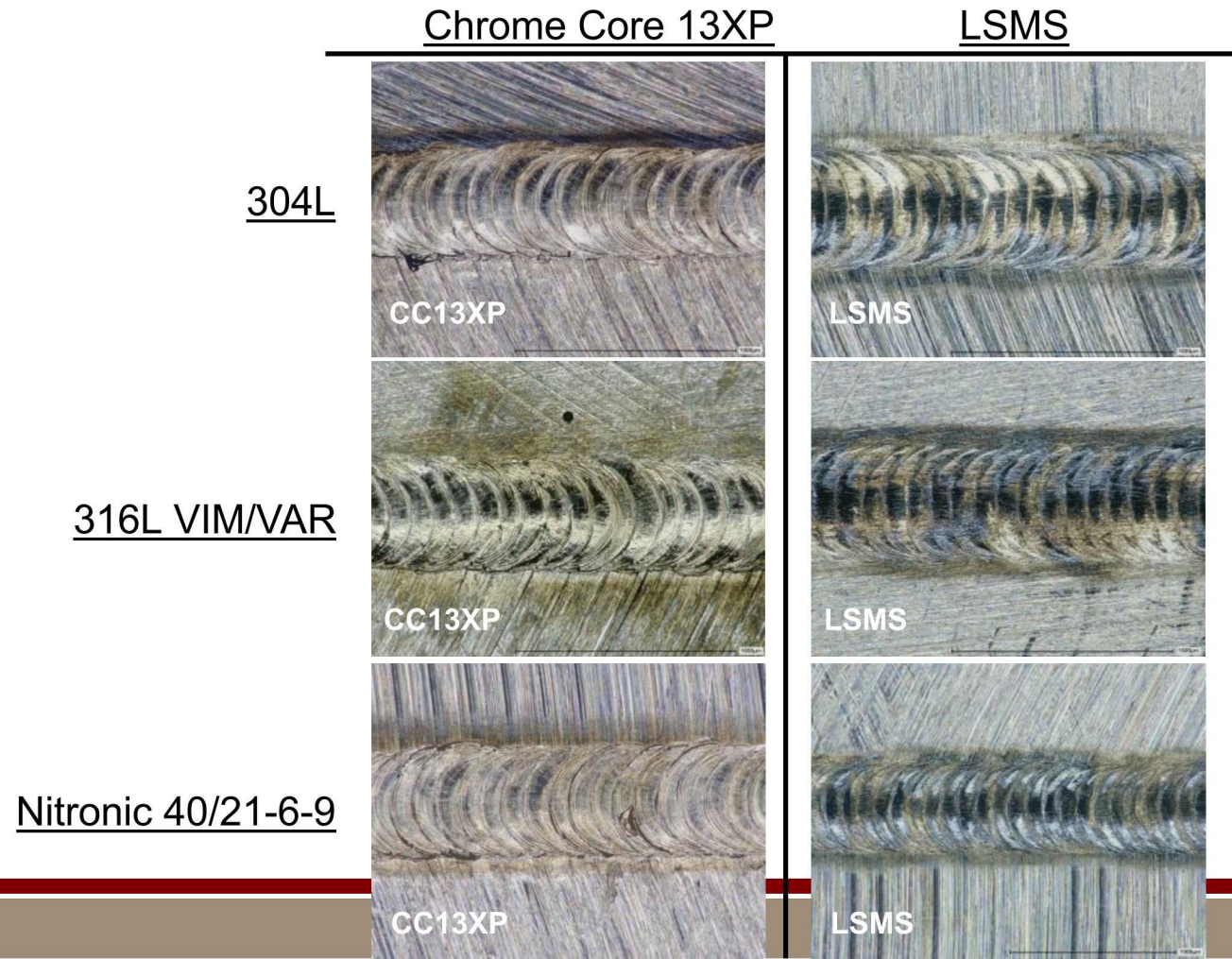
SEM Backscatter Electron Micrograph



Single-phase weld metal solidification with no terminal S-rich microconstituent

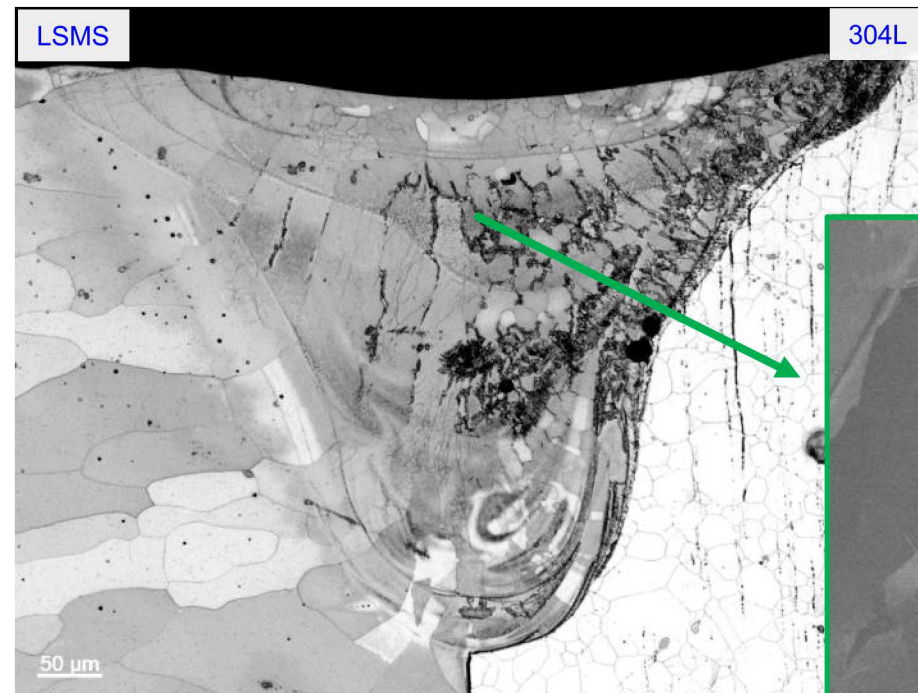
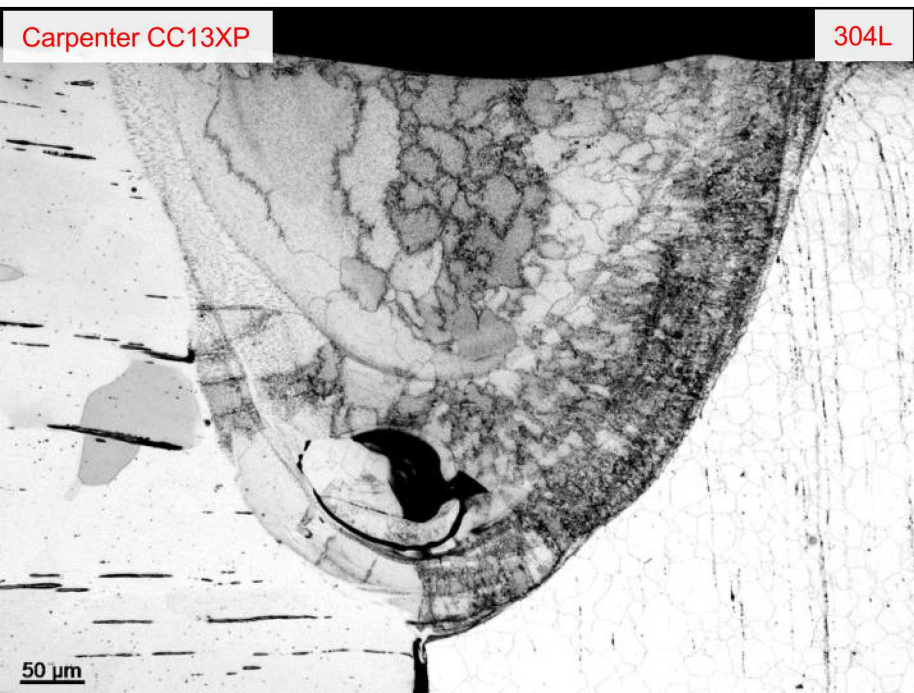
Weldability of LSMS opens new design options

- Dissimilar alloy joining of LSMS using laser welding enables new design freedom and reduces assembly complexity
- Various LSMS-austenitic stainless steel combinations have been evaluated



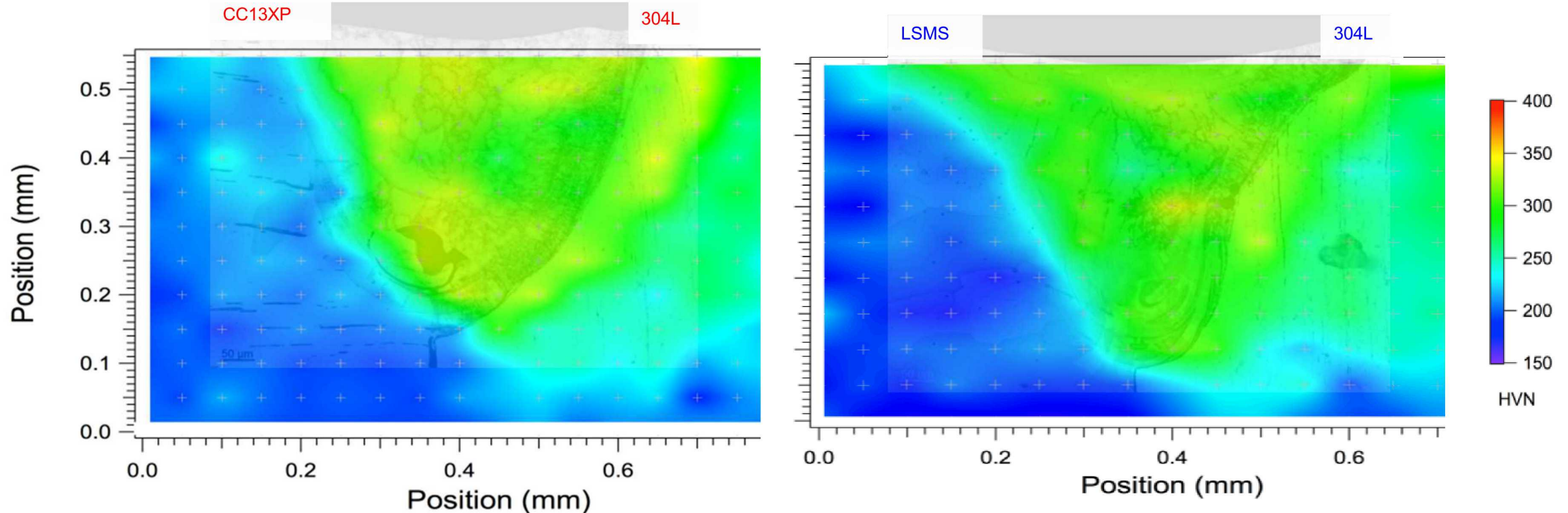
Example: Dissimilar 304L-LSMS laser weld

- CC13XP-304L combination carries intrinsic risk of solidification cracking
- Weld pool shape difference for LSMS vs. CC13XP due to effect of sulfur on surface tension driven fluid flow



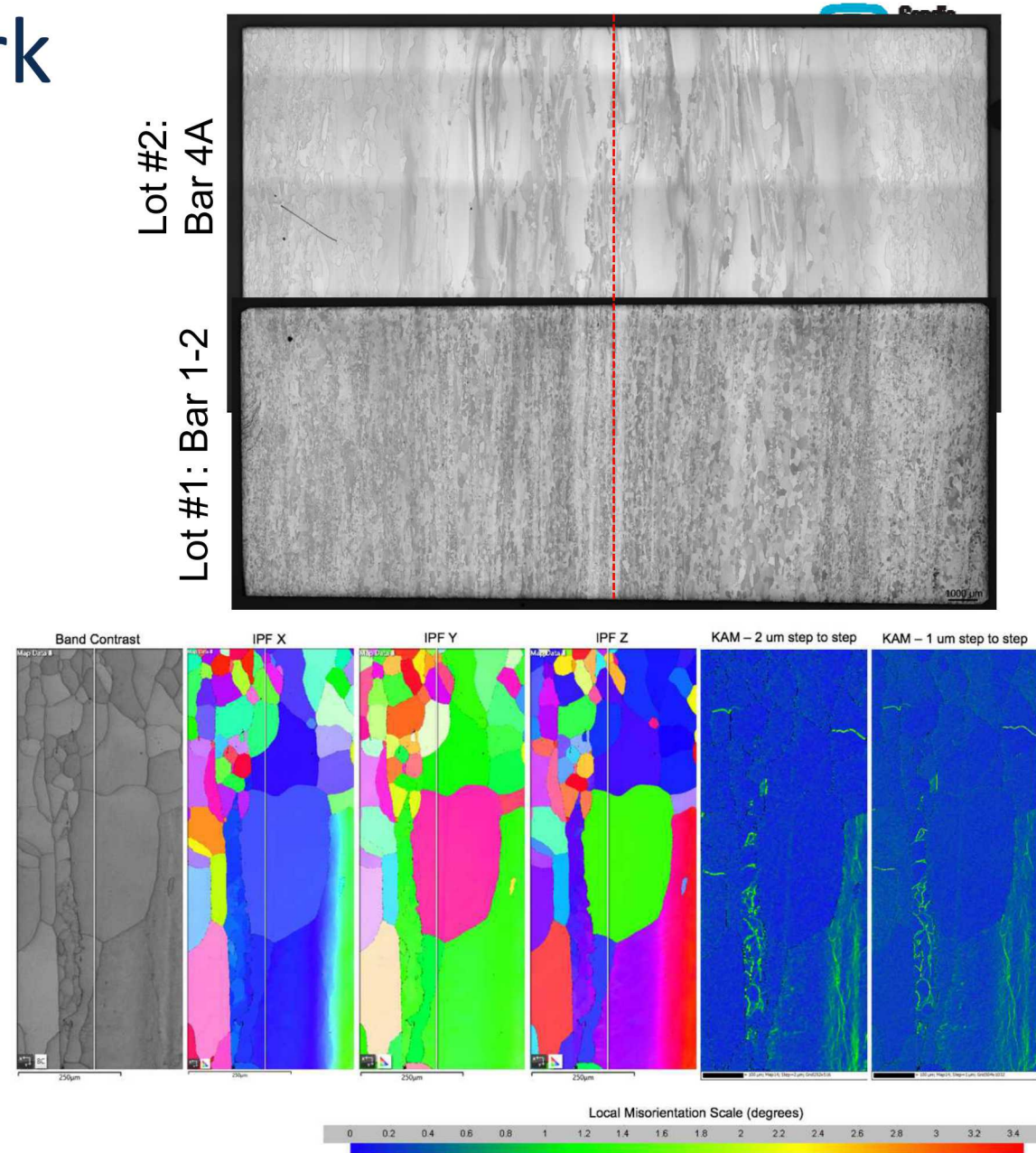
Microhardness Map Results: LSMS/CC13XP-304L

- Microhardness mapping shows expected mechanical property gradient in dissimilar weld
- Peak hardness remains 400 HV or lower—minimal mechanical property (ductility) degradation expected
- No significant HAZ softening measured for any dissimilar alloy combination.



Challenges and on-going work

- Lot-to-lot magnetic property variability necessitates additional work to strengthen process-structure-property relationships for LSMS.
- Local misorientation measurements (via high-acquisition rate EBSD) will be used to quantify dislocation density (stored energy) in LSMS as a function of processing parameters and annealing time/temperature.
- Production process optimization. E.g., hot working procedures (e.g., bar rolling vs. GFM) and/or introduction of cold-work prior to annealing



Concluding Remarks

- LSMS offers advantages over commercially-available equivalent (CC13XP) in aggressive environments where corrosion is a concern
 - Corrosion resistance improved via the elimination of MnS stringers
- Like other soft magnetic alloys, annealing of LSMS is used to drive recrystallization and grain growth
- Magnetic properties (B-H characteristics) do not appear to be highly sensitive to soak time at 850C.
 - Nominal heat treatment time for LSMS is 850C;4 hours
- Reduction of sulfur concentration in LSMS enables joining via welding. Welding of CC13XP carries risk of solidification cracking.
- LSMS can be laser welded to various dissimilar austenitic stainless steels. Joining of LSMS via welding enables new design concepts and can improve mechanical robustness.

Acknowledgements

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- Charlie Robino
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- Danny Maccallum
- Chris Finrock
- Sharon Shivley
- Mike Popper

BACKUP SLIDES

Second Development Lot of LSMS

- Repeatability was evaluated by requesting Lot #1 composition targets
- ~253# of finished 1" diameter bar delivered Sept '16. Entire lot magnetically heat treated (850° C, 3.5 hrs)
- Lot #2 bulk chemistry similar to original Lot #1

Element	Composition Range as per 7342522 (wt%)	LSMS Lot #1 (M723260) (wt%)	LSMS Lot #2 (M723866) (wt.%)
C	0.03 max.	0.009	0.01
P	0.03 max.	<0.005	<0.01
Si	1.20-1.80	1.27	1.47
Mo	0.50-1.00	0.78	0.78
Mn	0.50 max.	0.006	0.01
Cr	12.50-13.50	13.04	12.94
V	0.5-1.00	0.75	0.73
S	0.010 max.	0.003	0.003
N	report only	0.004	0.002
Ni	report only	0.01	0.008
Cu	report only	<0.01	<0.005
Co	report only	0.01	0.01
Al	report only	0.01	0.007
Fe	balance	balance	balance

LSMS Lot #2: Reported Magnetic and Mechanical Properties

- Further magnetic testing needed to characterize magnetic property anisotropy
- Reported mechanical properties similar to Lot #1 reported values

Magnetic Properties

	Sample	Applied Field, H (Oe)					DC Coercivity (Oe)
		2.0	3.0	5.0	30	200	
Flux Density, B (T)	LSMS Lot #1 (M723260): Ring	0.548	0.774	0.977	1.304	1.613	1.21
	LSMS Lot #1 (lot M723260): Bar	0.545	0.791	1.032	1.352	1.624	1.16
	LSMS Lot #2 (M723866): Ring	0.536	0.717	0.922	1.290	1.620	1.01
	LSMS Lot #2 (M72386): Bar	0.200	0.541	0.896	1.290	1.590	2.38
	Specification (7342522)	0.25 min.	0.52 min.	0.86 min.	1.30 min.	1.52 min.	2.0 max.

Mechanical Properties

	UTS (ksi)	YS (ksi)	Elongation (%)	Reduction in Area (%)
Specification (7342522)	60 min.	40 min.	30 min.	45 min.
LSMS Lot #1 (M723260)	69.5	44.2	37	82
LSMS Lot #2 (M723866)	66.5	46.3	38	81

Resultant Magnetic and Mechanical Properties

	Sample	Applied Field, H (Oe)					DC Coercivity (Oe)
		2.0	3.0	5.0	30	200	
Flux Density, B (T)	LSMS Lot #1 (lot M723260): <u>Ring</u>	0.548	0.774	0.977	1.304	1.613	1.21
	LSMS Lot #1 (lot M723260): <u>Bar</u>	0.545	0.791	1.032	1.352	1.624	1.16
	Specification (7342522)	0.25 min.	0.52 min.	0.86 min.	1.30 min.	1.52 min.	2.0 max.
	Carpenter CC13XP (heat: 734539)	0.268	0.528	0.921	1.400	1.630	1.93

Tested as per ASTM A773

	UTS (ksi)	YS (ksi)	Elongation (%)	Reduction in Area (%)
Specification (7342522)	60 min.	40 min.	30 min.	45 min.
LSMS Lot #1 (lot: M723260)	69.5	44.2	37	82
Carpenter CC13XP (heat: 734539)	68.0	46.3	34	62

Tested as per ASTM E8

- Magnetic and mechanical property testing was performed on material heat treated for ‘ideal’ magnetic properties (850°C for 3.5 hrs.)
- LSMS material demonstrates higher flux density (B) at low applied fields ($H \leq 3$ Oe) and nearly identical B at $H \geq 5$ Oe.
- No significant difference in measured values for ring vs. bar specimens
- DC coercivity for MWP considerably less than CC13XP reported value.
 - Need to obtain details for test procedure from KJS (external testing house)
- Mechanical properties for MWP nearly identical for CC13XP reported values.

