

Optimization of Advanced Steels for Cyclic Operation through an Integration of Material Testing, Modeling and Novel Component Test Validation

DE-FE002620, P.O.P. 9/3/15 to 3/30/18

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NETL Project Manager

Project Goals and Objectives

- To develop the needed microstructural processing and performance relationships and associated material models for specific constituents in fabricated weldments (such as the parent material, heat affected zone regions and weld metal),
- Apply these metallurgical relationships through modeling of a welded pressure bearing power plant component subjected to cyclic operational conditions under both mechanical and thermal loading, and
- Validate the model through novel structural feature and component tests.

Milestone Description	Completion Date
Task 1.0 – Kickoff Meeting	11/6/2015
Task 1.0 – Updated Project Management Plan	9/24/2015
Task 1.0 – Submit Final Report	6/30/2018
Task 2.0 – Materials and Processing	1/26/2016
Task 3.0 – Fabrication of Test Coupons	3/1/2016
Task 4.0 – Testing of optimized Grade 92 steel parent material and weldments	2/28/2018
Task 5.0 – Microstructural Evaluation of chosen material	3/31/2018
Task 6.0 – Design and Modeling of Component Test	2/19/2018
Task 7.1 – Conceptual design for a feature test of parent material and weldments under flexible operation	4/29/2016
Task 7.2 – Assemble and complete a check-out test on one experimental test frame for use in Phase 2 follow-on proposal work	1/31/2018

Outline

- Motivation for the Research
- Team Assembled, Plan, and Defining Test Conditions & Materials
- Experimental Approach
- Results and Ongoing Characterization
- Future Work and Summary

Motivation for Research

Today's 'Options' for State-of-the-Art HRSGs; Steam $\geq 600^{\circ}\text{C}$

Summary of Key Challenges facing OEMs [Not limited to]:

- ❑ Steamside Oxidation
- ❑ "Air" Oxidation (high moisture content in exhaust gas)
- ❑ Materials with High Creep Strength
- ❑ Materials with variable Creep Ductility
- ❑ Design by rule is inadequate to achieve the stated life and cycling objectives. Conversely, available Design by Analysis approaches for fatigue, creep or creep-fatigue vary significantly

Option 1a: Stainless Steels

Advanced grades (principally Super 304H) or 'traditional' 300 series "H" grades (principally 347H)

Dissimilar metal welds

Option 1b: Substitute stainless steel for lean Ni-base alloy

Lean Ni-base alloy options include HR6W, alloy 800H and potentially others

Dissimilar metal welds

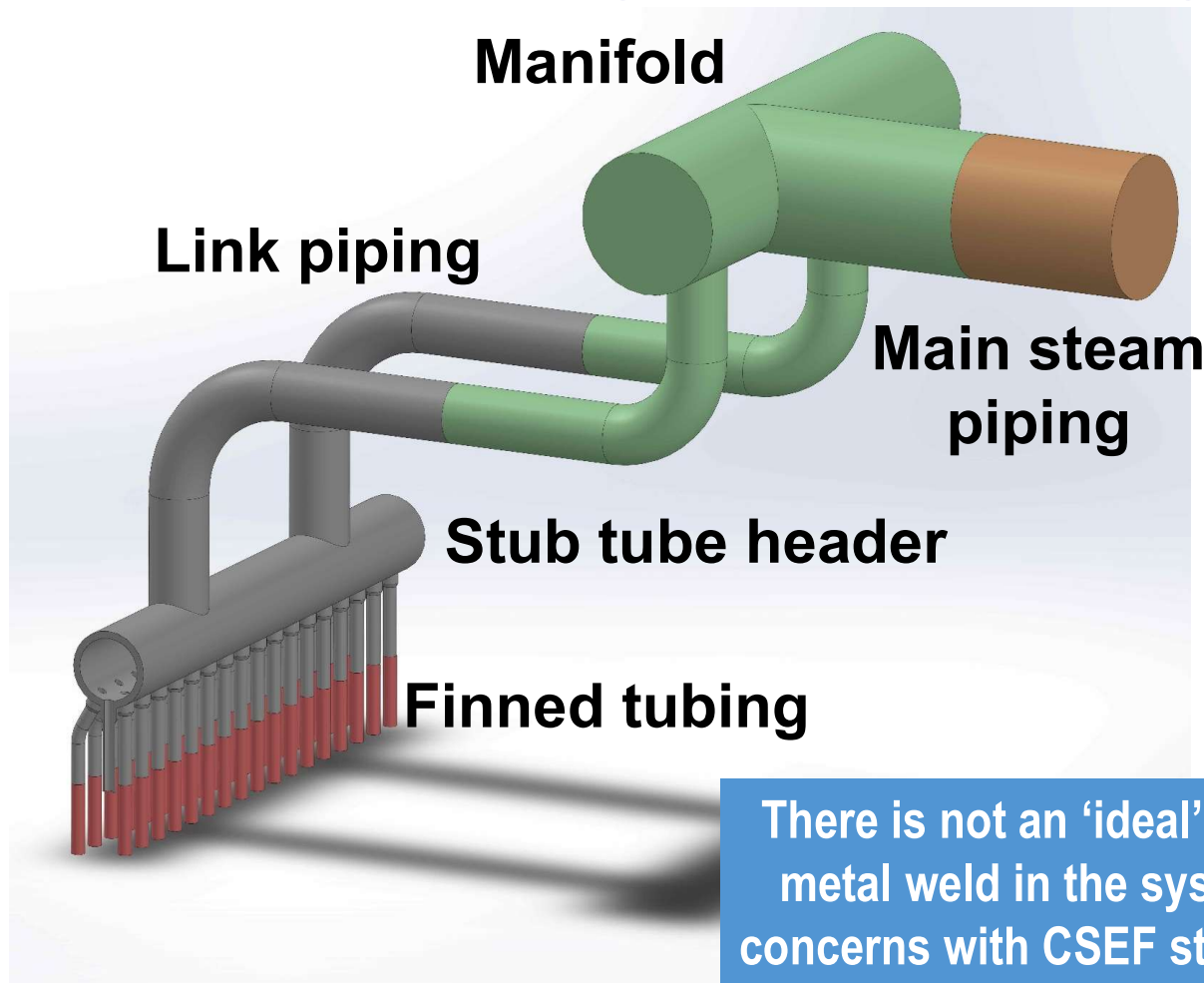
Option 2: 'More' Creep Strength Enhanced Ferritic Steels

Oxidation resistant 11Cr variants (i.e. THOR 115 and VM12SHC)

High creep strength 9%Cr variants (i.e. Gr. 92 and SAVE 12AD) and 'pushing the performance envelope' of Gr. 91/Gr. 92

Should avoid DMWs in flexible operation whenever possible

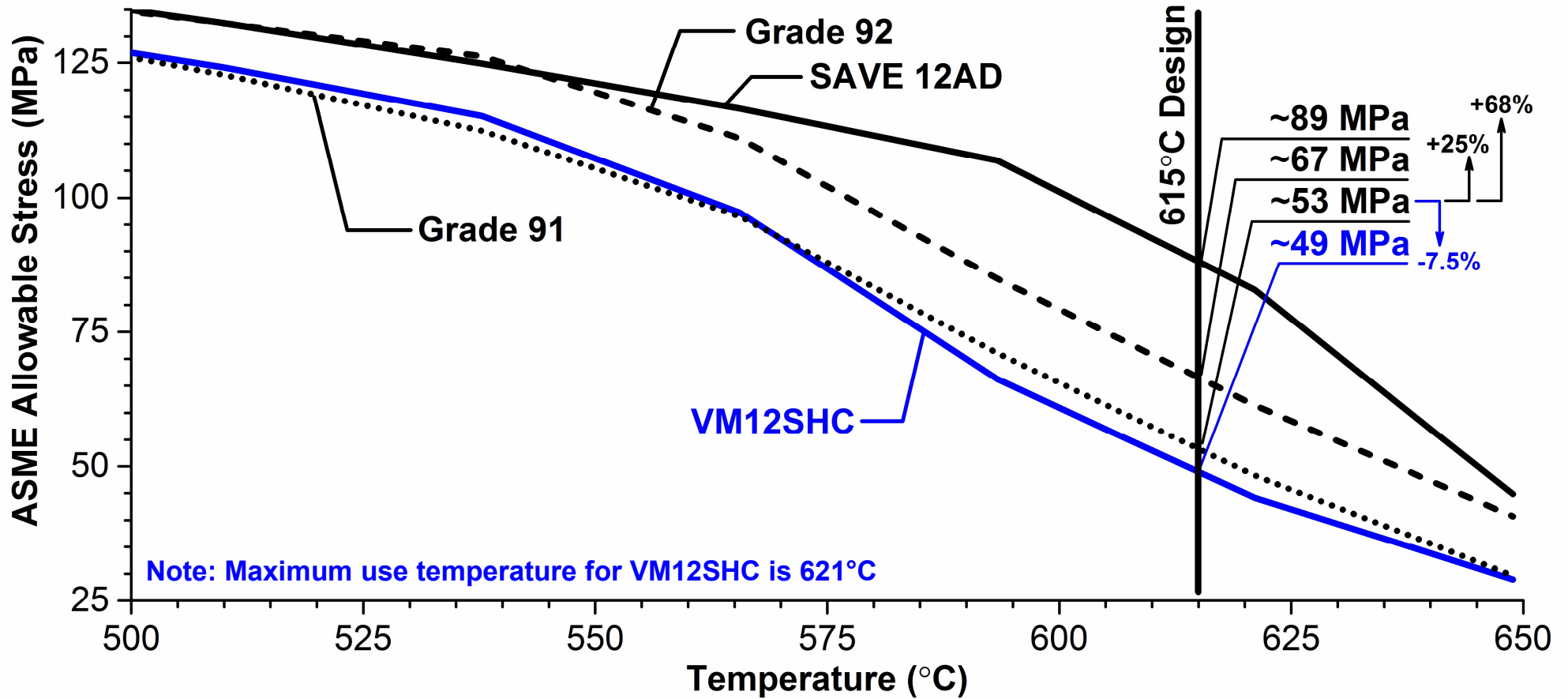
Materials Challenges: HRSG Configuration



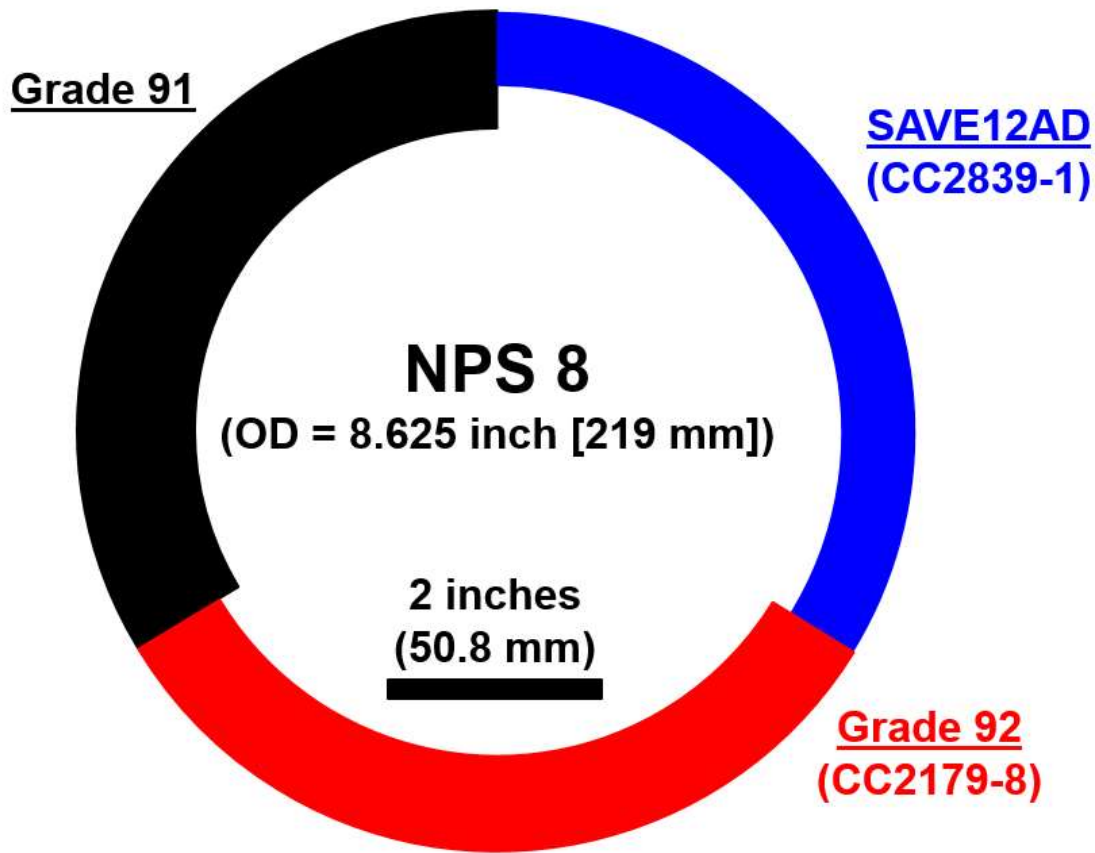
- Practical challenges
 - Where to place DMWs
 - Designing with stainless steels
 - Thermal-fatigue
 - Welding
 - Sensitization
 - Metallurgical risk (sigma phase evolution)

There is not an 'ideal' option for placement of a dissimilar metal weld in the system. Although there are significant concerns with CSEF steels, it may be the lesser of two evils

Stress Allowable Comparison for 9Cr and 12Cr Materials [Ref. SA-213 T91, CC2179-8, CC2781, CC2839]



Comparison of Required Wall Thickness for a NPS 8 Superheater Outlet Header



Material	Allowable Stress at 615°C (1140°F) (MPa)	Code required wall thickness (mm)
Grade 91	52.1	30.5
Grade 92 (CC2179-8)	64.7	25.5
Grade 93, SAVE12AD (CC2839-1)	88.6	19.5

Material selection for this application is a balance of oxidation resistance and allowable stress

And Cost / Value

There is still Significant Concern over Detailed Composition:

e.g. SAVE 12AD, ASME File: 013_1679_Background_Rev5

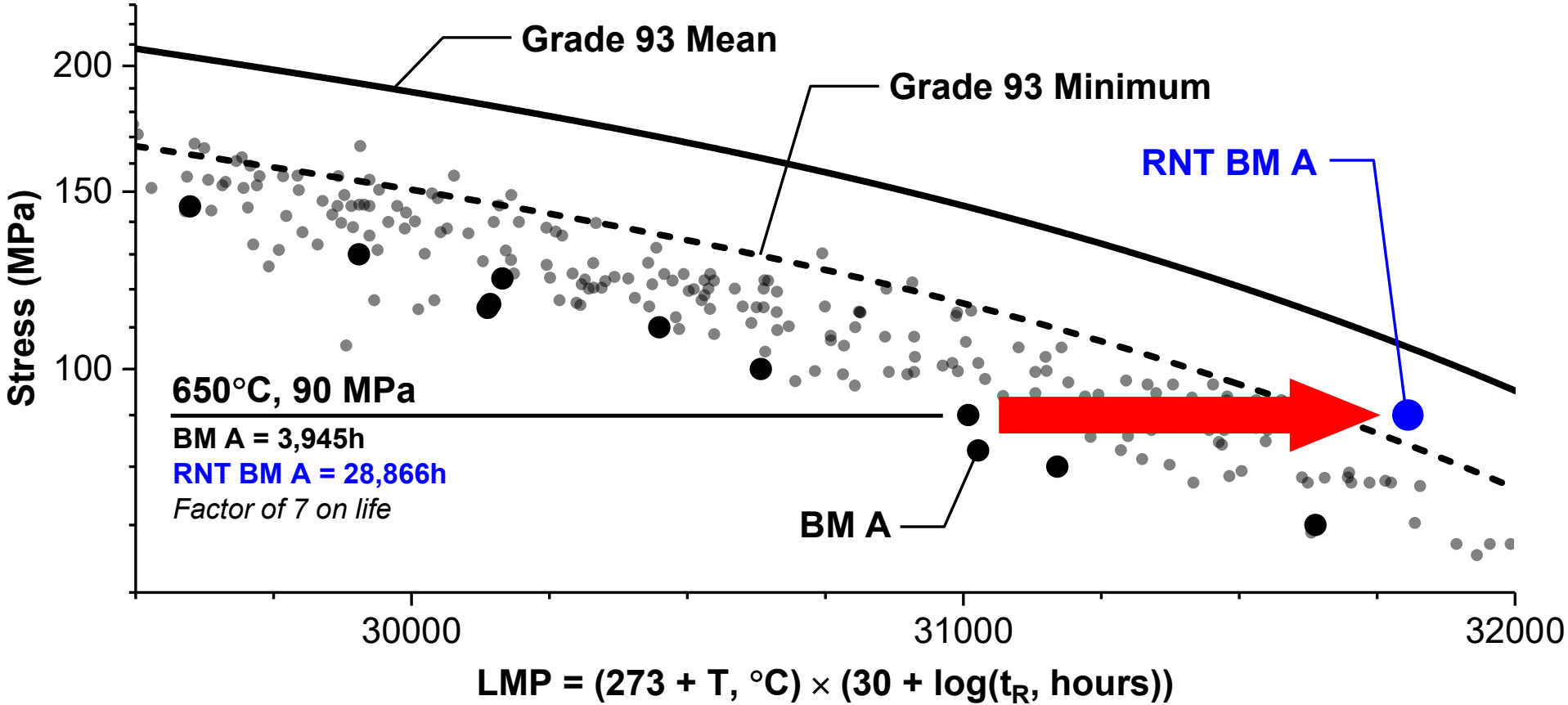
Heat		C	Si	Mn	P	S	Ni	Cr	W	Co
Heat 1		0.07	0.25	0.52	0.016	0.0010	-	8.91	2.99	1.04
Heat 2		0.08	0.24	0.52	0.011	0.0010	0.10	9.22	2.98	2.97
Heat 3		0.08	0.25	0.50	0.010	0.0010	0.10	9.00	3.00	2.98
Heat 4		0.08	0.24	0.51	0.012	0.0010	0.10	8.77	3.01	3.00
Heat 5		0.08	0.25	0.50	0.012	0.0010	0.10	8.53	2.99	2.99
Heat 6		0.08	0.24	0.51	0.011	0.0010	0.10	8.99	2.99	3.27
Heat 7		0.08	0.26	0.51	0.009	0.0010	0.11	9.11	2.89	2.63
Heat 8		0.08	0.25	0.52	0.009	0.0005	0.11	9.17	2.89	2.62
Heat 9		0.08	0.26	0.52	0.009	0.0010	0.12	8.86	2.88	3.02
Heat 10		0.08	0.24	0.53	0.011	0.0008	0.10	9.27	2.82	2.81
Heat 11		0.08	0.29	0.49	0.011	0.0008	0.09	9.06	2.96	2.99
Heat 12		0.08	0.23	0.49	0.013	0.0009	0.08	9.12	2.90	3.06
Heat 13		0.08	0.27	0.50	0.013	0.0010	0.11	9.08	2.88	3.00
Heat 14		0.08	0.24	0.52	0.014	0.0012	0.08	9.24	2.91	3.07
Specification	min.	0.05	0.05	0.20				8.50	2.0	1.0
	max.	0.13	0.50	0.70	0.020	0.010	0.50	9.50	3.5	3.5
	Min	0.07	0.24	0.49	0.009	0.0008	0.08	8.53	2.88	1.04
	Max	0.08	0.29	0.53	0.016	0.0012	0.12	9.24	3.01	3.07

**There is still Significant Concern regarding Processing details:
e.g. SAVE 12AD, ASME File: 013_1679_Background_Rev5**

Steel	Heat	Product Form	Dimensions (mm)	Heat Treatment
S1	Heat1	Plate	t15	1150°C x1h AC → 780°C × 1h AC
S2	Heat2	Plate	t15	1150°C x1h AC → 780°C × 4h AC
S3	Heat2	Plate	t15	1150°C x2h AC → 780°C × 4h AC
S4	Heat3	Plate	t15	1150°C x1h AC → 780°C × 4h AC
S5	Heat3	Plate	t15	1150°C x2h AC → 780°C × 4h AC
S6	Heat4	Plate	t15	1150°C x1h AC → 780°C × 4h AC
S7	Heat4	Plate	t15	1150°C x2h AC → 780°C × 4h AC
S8	Heat5	Plate	t15	1150°C x1h AC → 780°C × 4h AC
S9	Heat5	Plate	t15	1150°C x2h AC → 780°C × 4h AC
S10	Heat6	Plate	t15	1150°C x1h AC → 780°C × 4h AC
S11	Heat6	Plate	t15	1150°C x2h AC → 780°C × 4h AC
S12	Heat7	Plate	t25	1150°C x1h AC → 780°C × 4h AC
S13	Heat8	Plate	t25	1150°C x1h AC → 780°C × 4h AC
T1	Heat9	Tube	38OD × 8.8WT	1150°C × 10min AC → 780°C × 2h AC
T2	Heat10	Tube	80OD × 20WT	1150°C x1h AC → 780°C × 4h AC
T3	Heat11	Tube	45OD × 8.5WT	1150°C × 10min AC → 780°C × 3h AC
P1	Heat12	Pipe	350OD × 50WT	1150°C x1h AC → 780°C × 3h AC
P2	Heat13	Pipe	350OD × 40WT	1150°C × 30min AC → 780°C × 6h AC
P3	Heat14	Pipe	350OD × 40WT	1150°C × 30min AC → 780°C × 6h AC

Proposed range = 1080 to 1170°C

If both the Composition & Processing are well selected; Grade 92 can achieve creep performance within the scatter-band for SAVE12AD



Many 9% - 11%Cr CSEF Steels have been Proposed, Challenge to Optimize Composition/Processing/Heat Treatment

- CSEF steels include:
 - Grade 91
 - NF616 (Grade 92)
 - HCM12A (Grade 122)
 - COST alloys (CB2/FB2)
 - SAVE12, SAVE12AD (Grade 93)
 - VM12, VM12SHC
 - THOR 115
 - Etc.

The emphasis should be to produce a material which exhibits:

- Consistent performance
- Easily formed
- 'Convenient' heat treatment
- Easily fabricated
- Minimal alloying
- Well-understood

The alloys to the left and after 'Grade 91' exhibit none-of-the-above

Code Implications – Reduce Variability

- New material development requires very large investment in time and money.
- Refinement/optimization of Gr. 92 (or Gr. 91) steels through application of a focus on **relevant** tests on **well pedigreed** steels, offers significant **benefits**
- Knowing parent metal performance under simple laboratory test conditions is NOT sufficient to **validate models**, sensible commercial use requires understanding :
 1. **Fabrication heterogeneity (metallurgical notches)**
 2. **Design details (mechanical notches)**
- **Stress state effects** (changing stress state influences both creep life & ductility):
 - Uniaxial stress loading: **notch bar** creep, simulated HAZ tests, feature cross-weld tests and
 - Multiaxial stress loading: end load + pressure such as in tubes & vessels and
 - Consideration of Case Studies from selected end-use applications - including flexible operation in HRSGs which results in through-wall ΔT – to establish performance for critical components

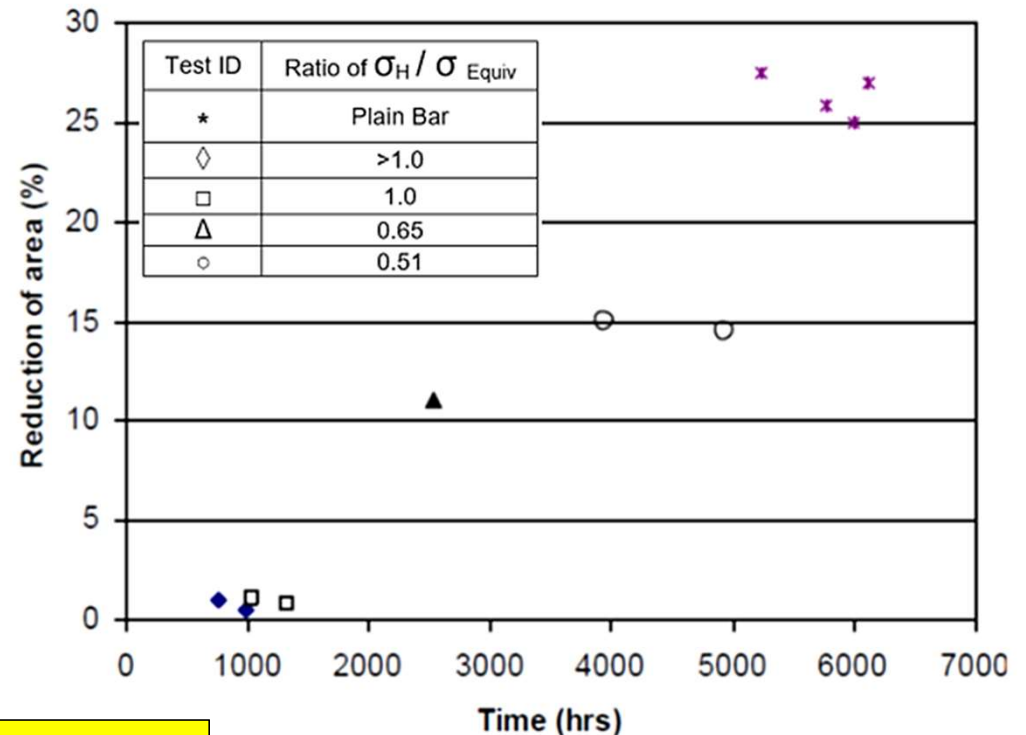
Material Data Needs to Support Design and Analysis

ASME and International Perspectives of Issues

Relationship between Reduction of Area & creep life for tests on 2¼Cr1Mo low alloy steel performed at 550°C on plain bar & notched bar specimens.

Different notch geometries were used to develop different stress states as defined using a triaxiality factor, e.g.

$$TF = \frac{\sigma_1 + \sigma_2 + \sigma_3}{\sigma_m}$$



Creep Life and Ductility are functions of constraint

O. Kwon, CW Thomas and D. Knowles, "Multiaxial stress rupture behaviour and stress-state sensitivity of creep damage distribution in Durehete 1055 and 2.25Cr1Mo steel", Int J of Pressure Vessels and Piping 81(6), 2004, pp 535-542

In-service Damage is a Function of Three Key Factors

■ Damage

- Measure = creep ductility [Elongation or reduction of area]
- Fundamental concept = Void nucleation
- Key microstructure features = Inclusions/ intermetallics

■ Deformation

- Measure = creep strength [time to failure, min creep rate, etc.]
- Fundamental concept = Void growth
- Key microstructure features = Particles on grain boundaries

■ Stress State

- Measure = Equivalent versus principal stress controlled damage
- Key microstructure features = distribution/extent of damage in carefully controlled tests which introduce multiaxial stress state

Systematic Evaluation of the Material Design Envelope

Steel	Melting	Processing	Final Heat Treatment	Composition
Gr. 91	<ul style="list-style-type: none"> <input type="checkbox"/> Understanding the influence of Ca-addition and optimization <input type="checkbox"/> Reduction in overall inclusion content <input type="checkbox"/> Desulphurization 	<ul style="list-style-type: none"> <input type="checkbox"/> Manufacture of pipe and influence hot-work has on performance <input type="checkbox"/> Billet-charging prior to forming 	<ul style="list-style-type: none"> <input type="checkbox"/> High temperature normalization, defined as 1080 to 1125°C <input type="checkbox"/> Cooling rate, such as in oil or water 	<ul style="list-style-type: none"> <input type="checkbox"/> Boron addition up to ~0.001 wt. % <input type="checkbox"/> Reduction in tramp elements <input type="checkbox"/> Reduction in S, Al, Ni <input type="checkbox"/> Ce addition
Gr. 92	<ul style="list-style-type: none"> <input type="checkbox"/> Optimization of Al-addition for deoxidation <input type="checkbox"/> Better understanding of continuous casting process for billet manufacturing 		<ul style="list-style-type: none"> <input type="checkbox"/> High temperature normalization, defined as 1125 to 1175°C <input type="checkbox"/> Cooling rate, such as in oil or water 	<ul style="list-style-type: none"> <input type="checkbox"/> N reduction to 0.010 wt. % <input type="checkbox"/> Reduction in tramp elements <input type="checkbox"/> Reduction in S, Al, Ni <input type="checkbox"/> Ce addition

- In all cases, the goal is three-fold:
 - Reduce material variability within a manageable specification
 - Increase creep ductility, ideally $\geq 70\%$ ROA under uniaxial smooth bar tests for parent material and for long-term $\leq 625^\circ\text{C}$ (1157°F)
 - Increase creep strength (e.g. to reduce influence of ΔT in component operation)

Team Assembled, Plan, and Defining Test Conditions & Materials

Tasks

- Task 2.0 – P92 Alloy Procurement and Processing [**Wyman**]
- Task 4.0 – Laboratory Scale Creep, Creep and Thermal Cycling Testing of P92 Samples [**EPRI**]
- Task 5.0 – Microstructural Evaluation of Initial Material, Heat Treatments and as-Tested Samples [**EPRI**]
- Task 6.0 – Development of Constitutive Equations, Creep-Fatigue Models and Design of a Phase II Pressure Vessel Component Test [**Structural Integrity Associates**]
- Task 7.0 – Design and Fabrication of a Structural Feature Scale Creep-Fatigue Test [**ORNL**]

Grade 92 Material

Analysis	C	Mn	P	S	Si	Ni	Cr	Mo	V
Cert	0.10	0.49	0.013	0.002	0.28	0.18	8.79	0.41	0.202
Ind. Analysis	0.084	0.47	0.008	0.0013	0.238	0.17	8.693	0.43	0.192
EPRI Rec.		0.30-0.50	<0.020	<0.005	0.20-0.40	<0.20			
Analysis	Cu	Al	As	Sn	W	B	Sb	Nb	N
Cert	0.18	0.005	0.007	0.011	1.77	0.0029	0.001	0.069	0.0418
Ind. Analysis	0.152	0.002	0.004	0.007	1.86	0.0023	0.0012	0.064	0.0480
EPRI Rec.	<0.10	<0.010	<0.010	<0.010			<0.003		

- Starting material = Grade 92; USA-sourced
 - Section size = 508 mm (20 inch) OD X 134 mm (5.27 inch) WT
 - As-received (1065°C target/air cool + 775°C/air cool)
 - **Improved/Optimized (1125°C minimum/oil quench + 775°C/air cool)**

Test Program – Emphasis on Relevance

- Carefully selected, well pedigreed steel
- Smooth bar creep (for database comparison)
 - Parent metal
 - Simulated HAZ ($T_{\text{peak}} \sim 900^{\circ}\text{C}/1\text{m}/\text{AC} + \text{PWHT}$)
- Parent metal notch bar creep (multiaxial stress state)
 - *Including a strict Code of Practice to ensure results are consistent*
- Feature type cross-weld creep (multiaxial stress state)
- Sequential testing to separate creep, fatigue and tensile damage mechanisms
 - Fatigue + creep
 - Creep + tensile



Evaluation of all samples to define deformation-damage-stress state effects

Results and On-going Studies

Notch Bar Creep Test Results

Sample	Temp.		Stress		Rupture limits, hrs.		Status*	Metallography
	°F	°C	ksi	MPa	Min	Max	hours	
NB-1a	1202	650	27.6	190		600	177	Yes
NB-2a	1202	650	24.7	170		1,000	509	Yes
NB-3a	1202	650	21.8	150		4,000	1,110	Yes
NB-4a	1202	650	18.1	125	400	12,000	2,660	Yes
NB-5a	1202	650	16.0	110	2,000	16,000	5,032	
NB-5b	1202	650	16.0	110	2,000	16,000	4,965	Yes

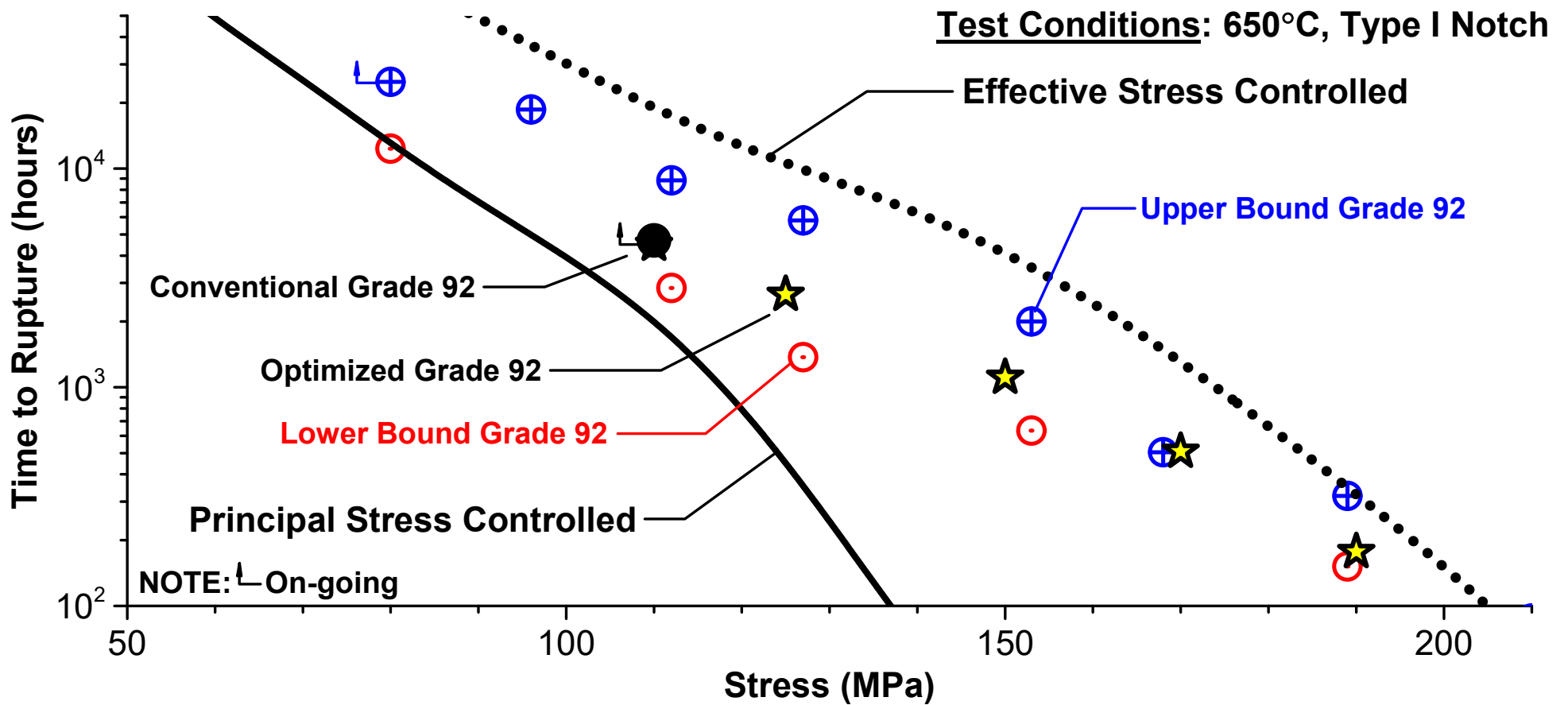
- **“a” Material = ‘Opt. Gr. 92’**

- DOE P92 material
- Normalization = 1150°C/1h/OQ
- Tempering = 775°C/3h/AC

- **“b” Material = ‘Conv. Gr. 92’**

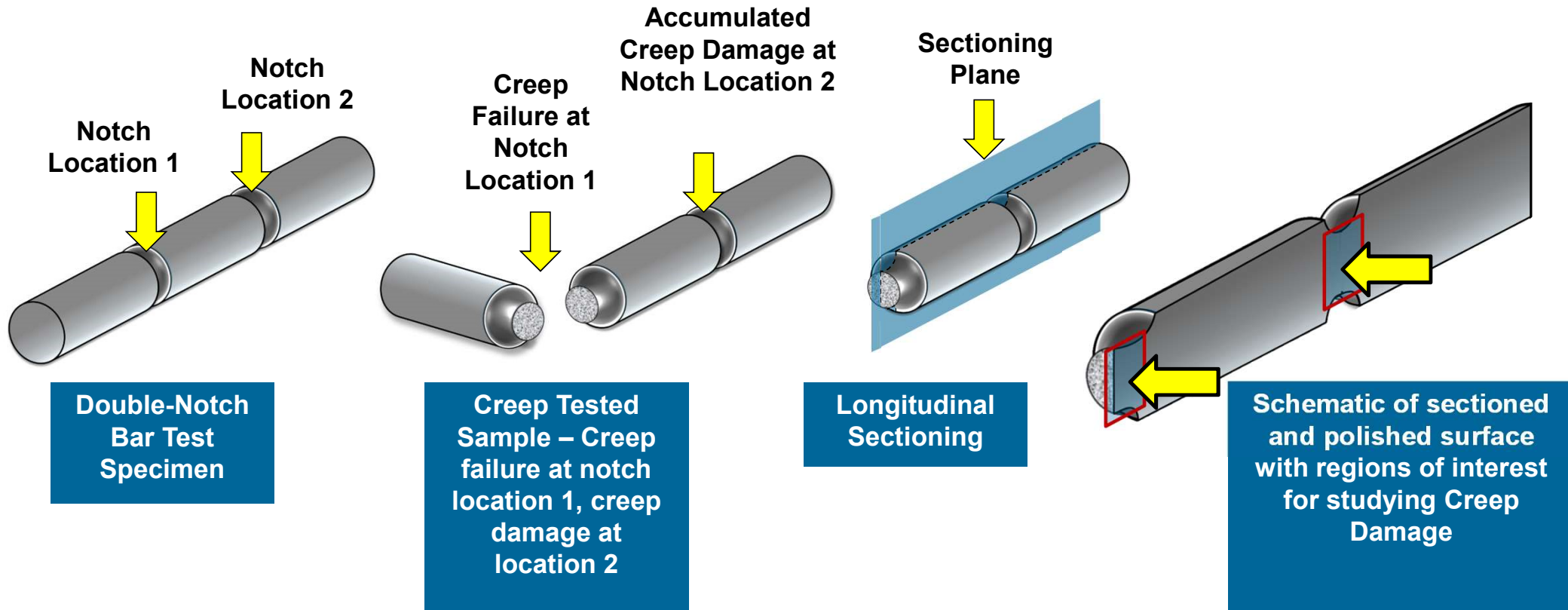
- DOE P92 material
- As-received condition where
 - Normalization = 1065°C/2.75h/AC
 - Tempering = 775°C/5.5h/AC

Comparison of Current Data to Grade 92 EPRI Database

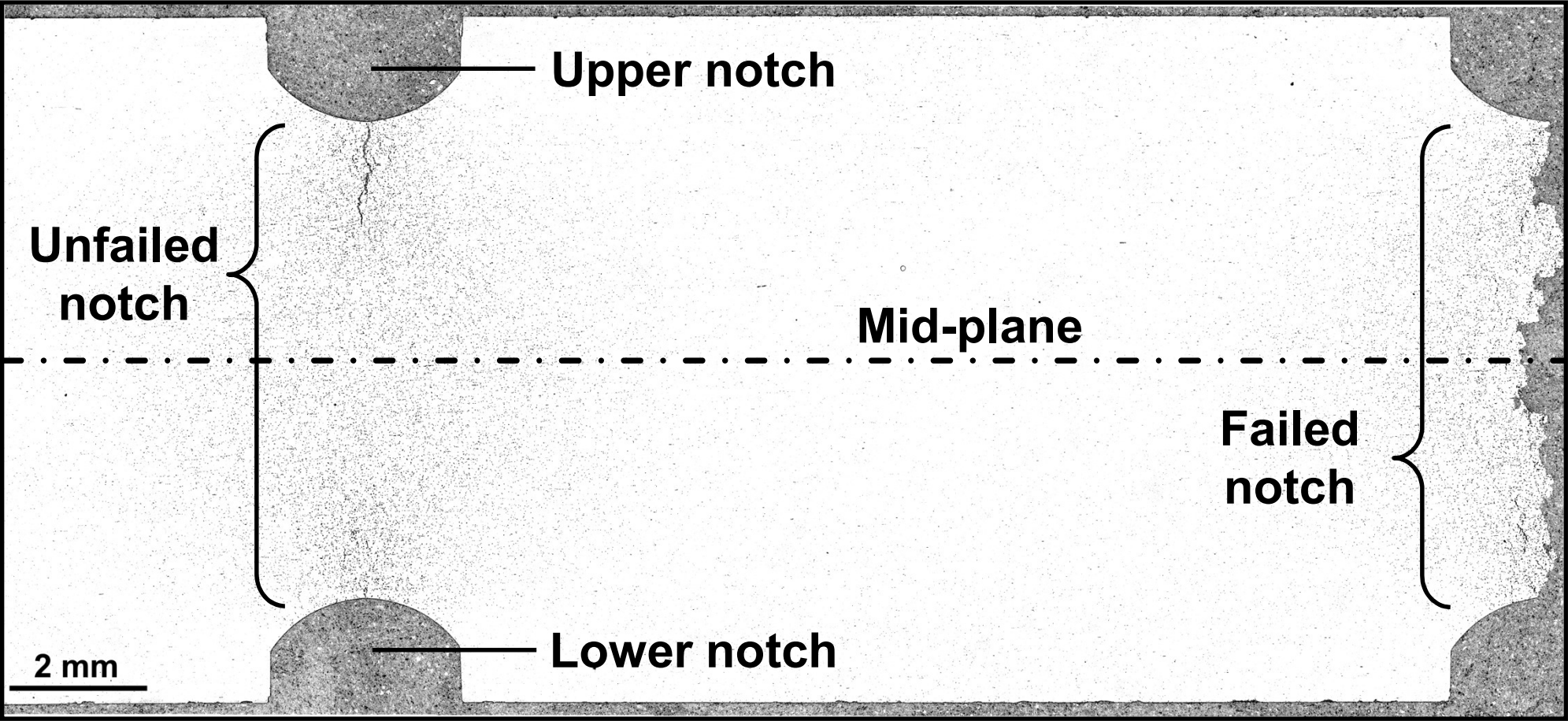


Notch Bar Test

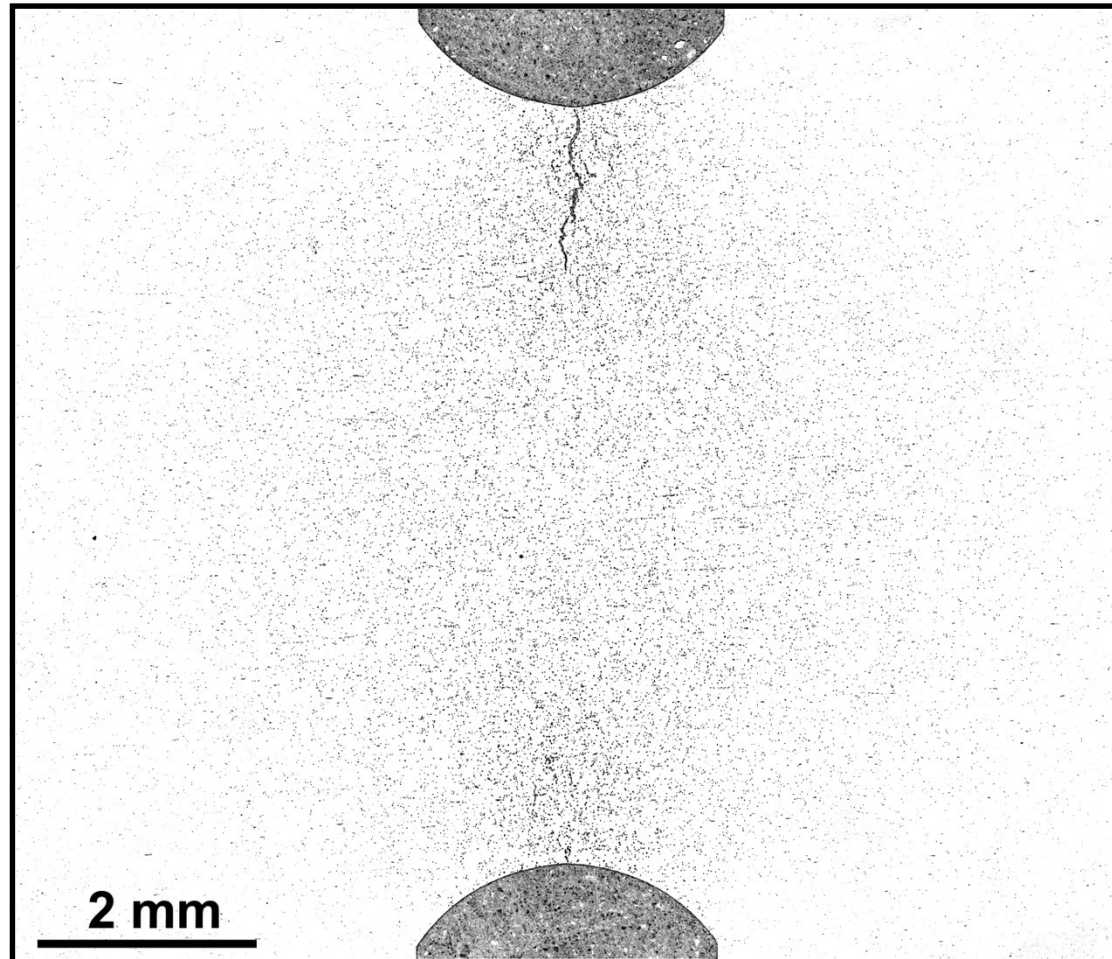
Sample Preparation for Metallographic Analysis



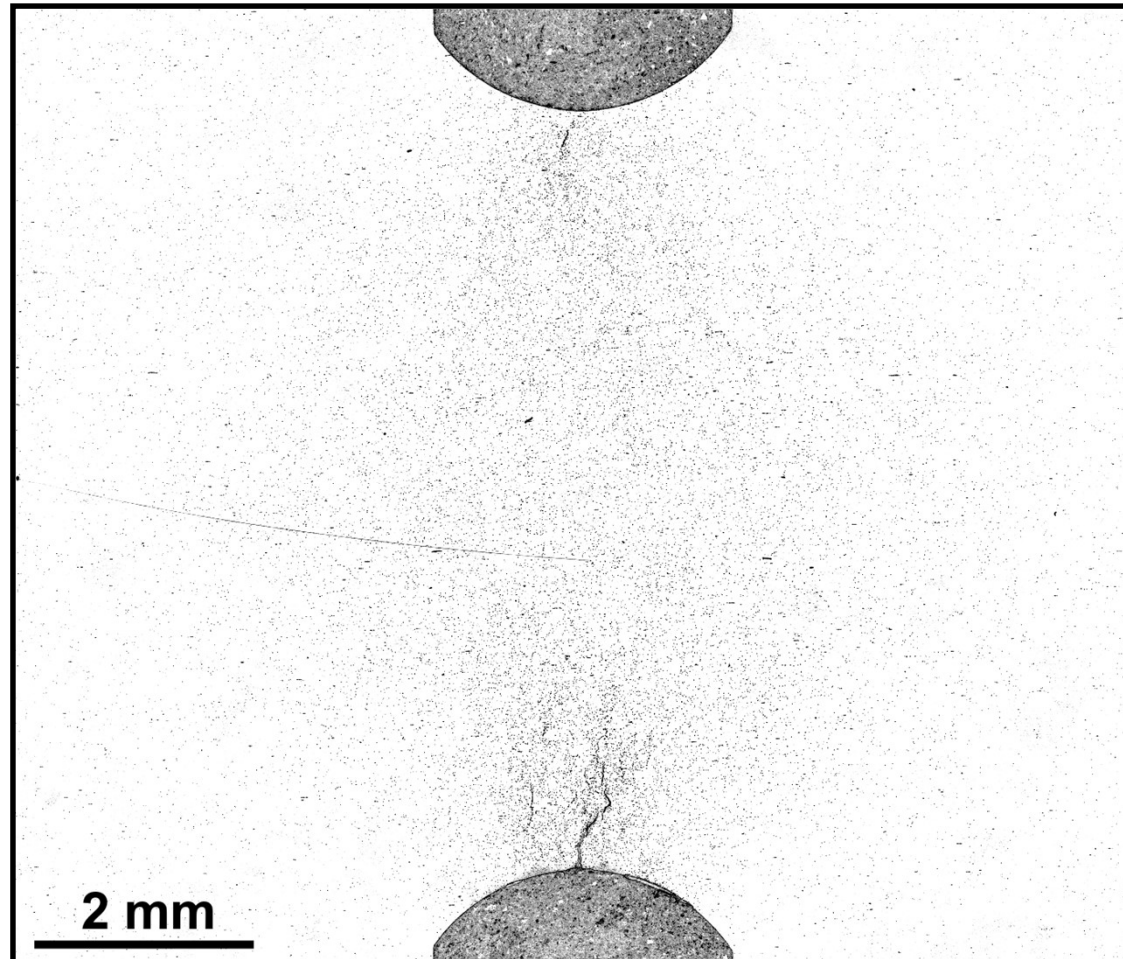
Example of Mounted Sample (NB-5b shown)



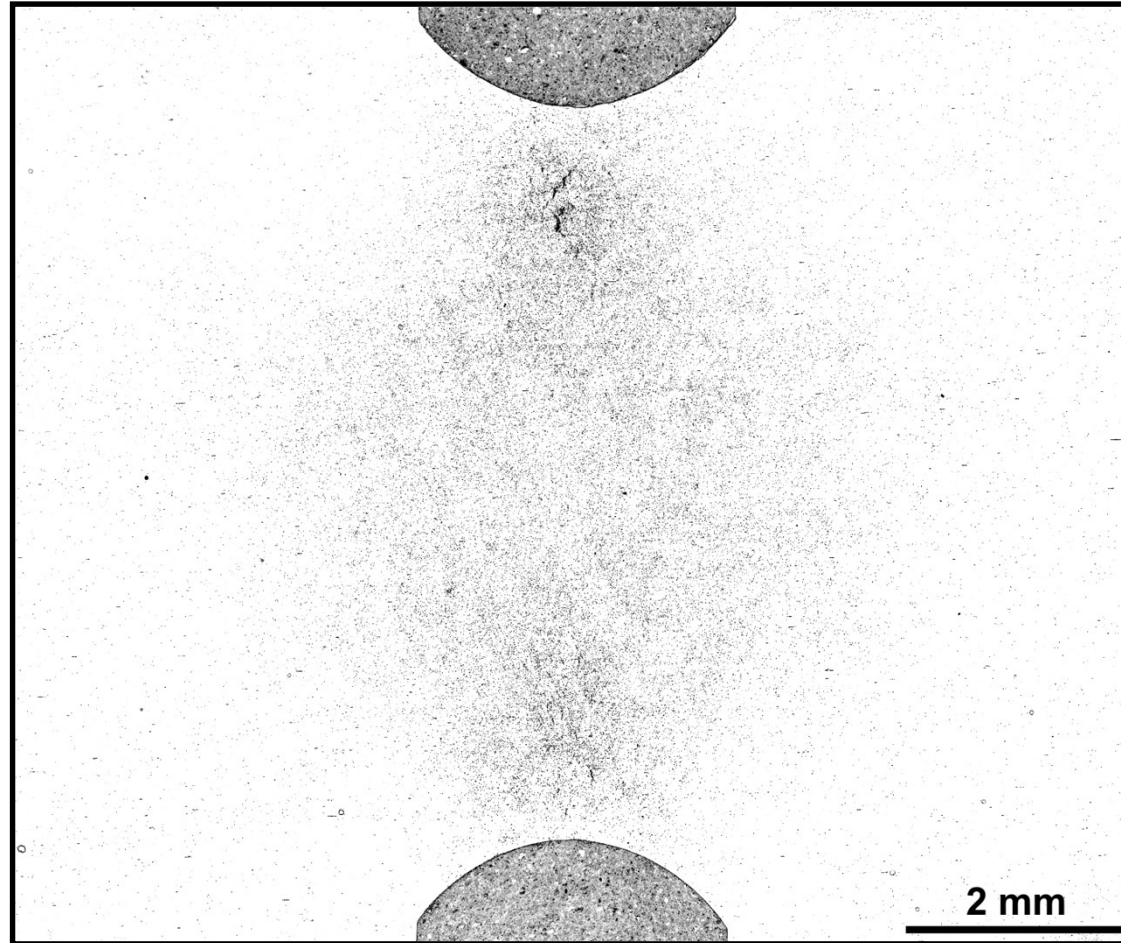
NB-5b; 650°C, 110 MPa, 4,965h



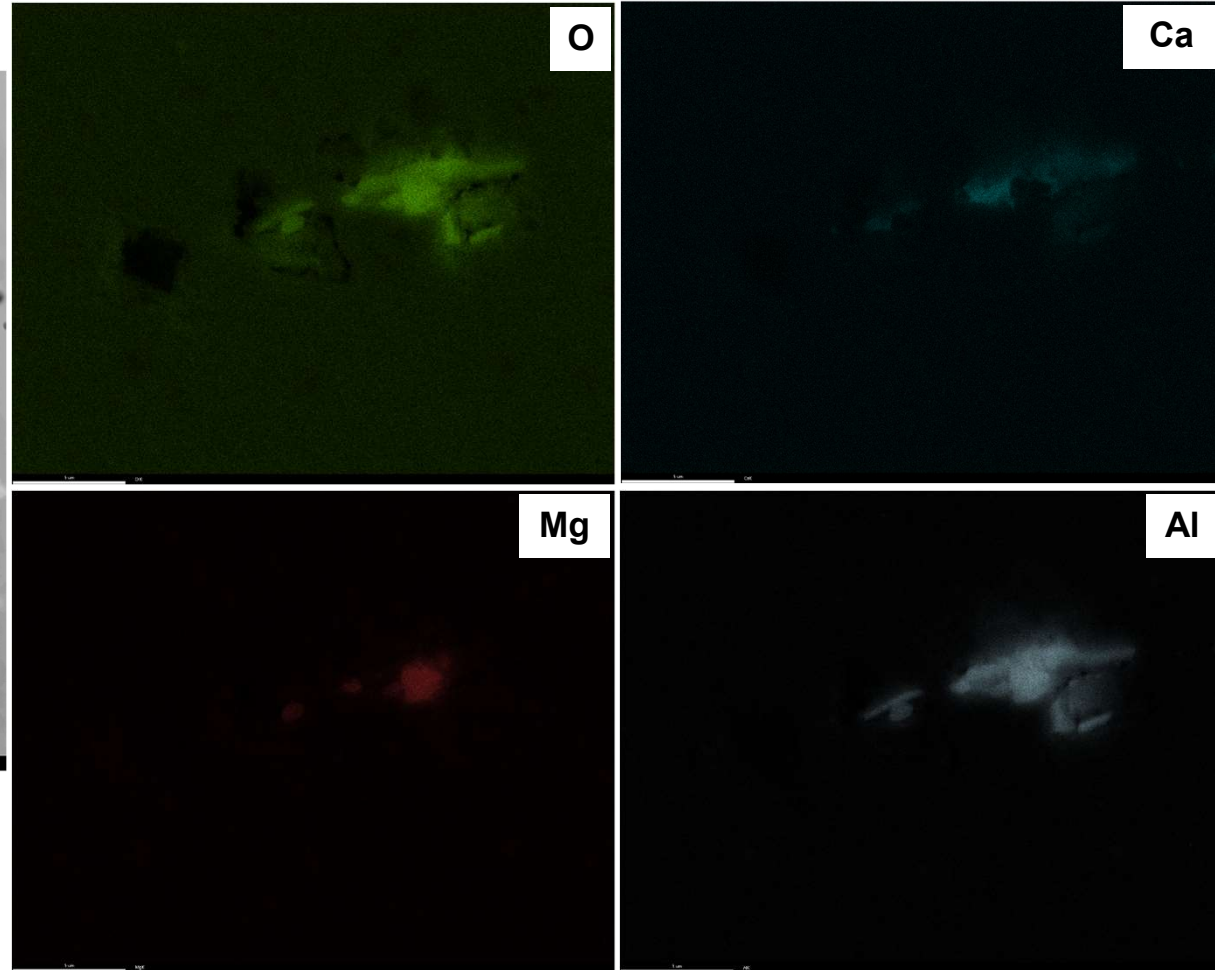
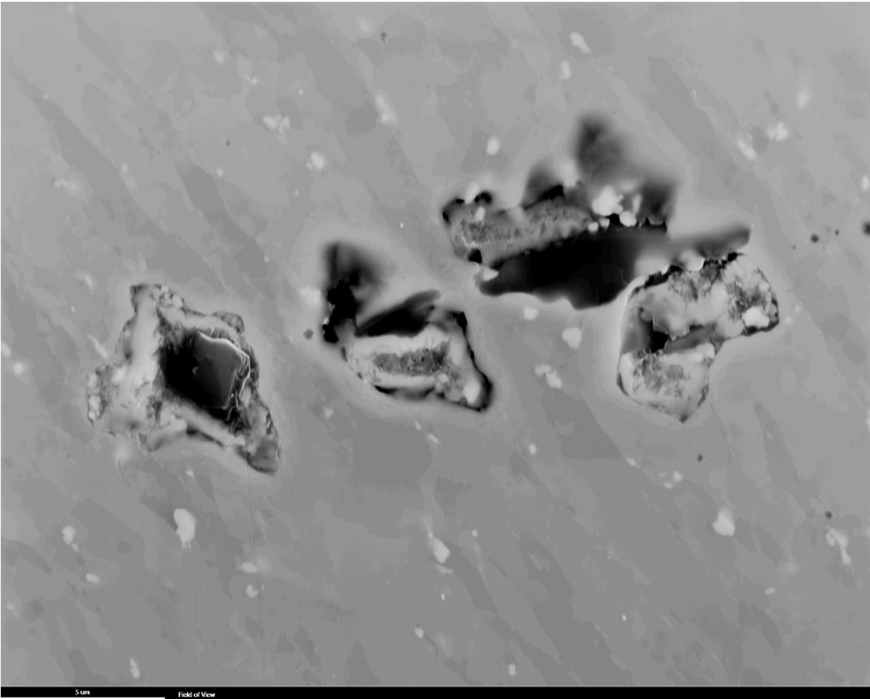
NB-4a; 650°C, 125 MPa, 2,660h



NB-1a; 650°C, 190 MPa, 177h



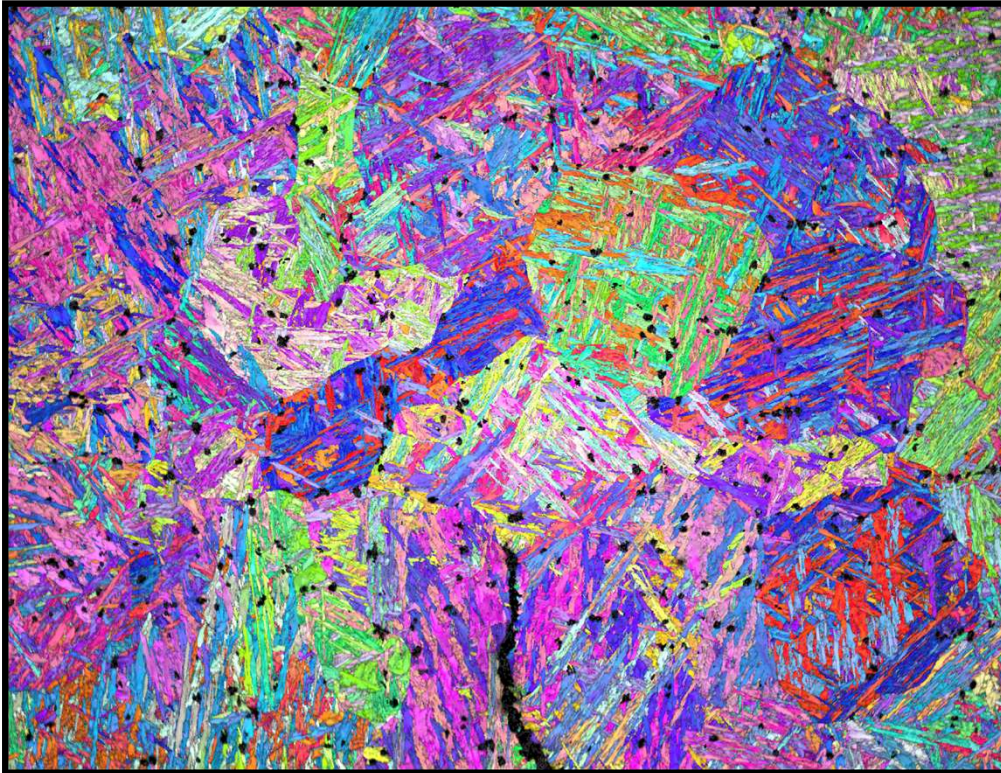
EDS Analysis Spot 1



- Calcium + magnesium aluminates; **inclusions are important to damage nucleation!**

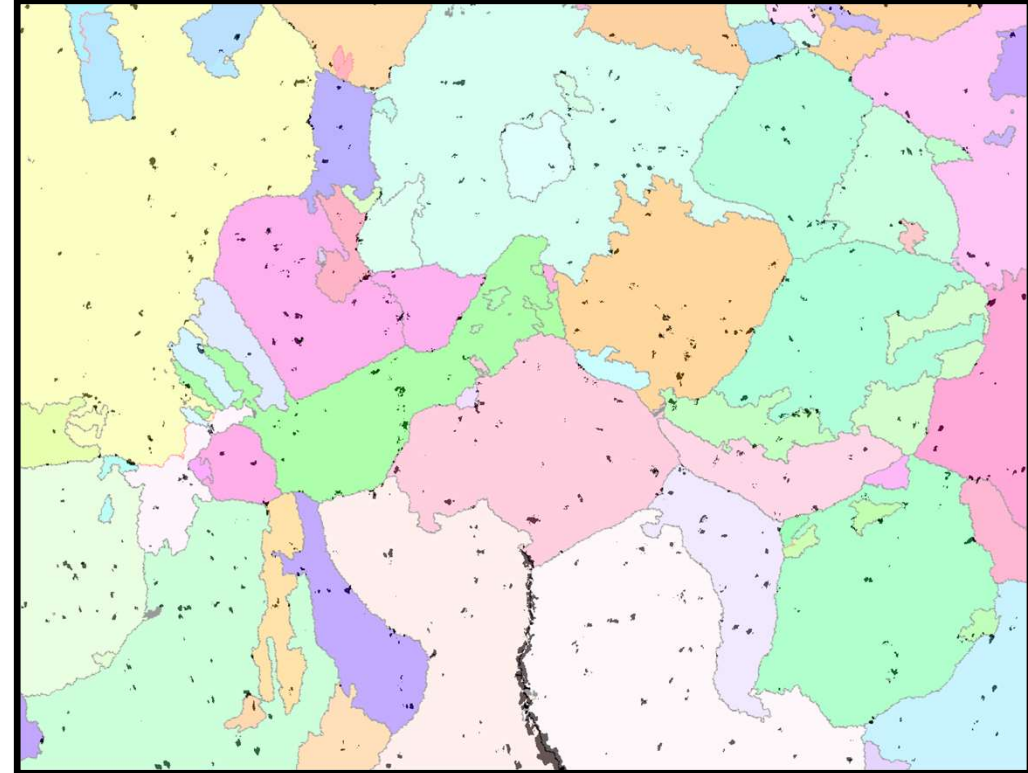
Prior Austenite Grain Reconstruction

Creep cavitation does not appear to preferentially occur at prior austenite GBs



200 μ m

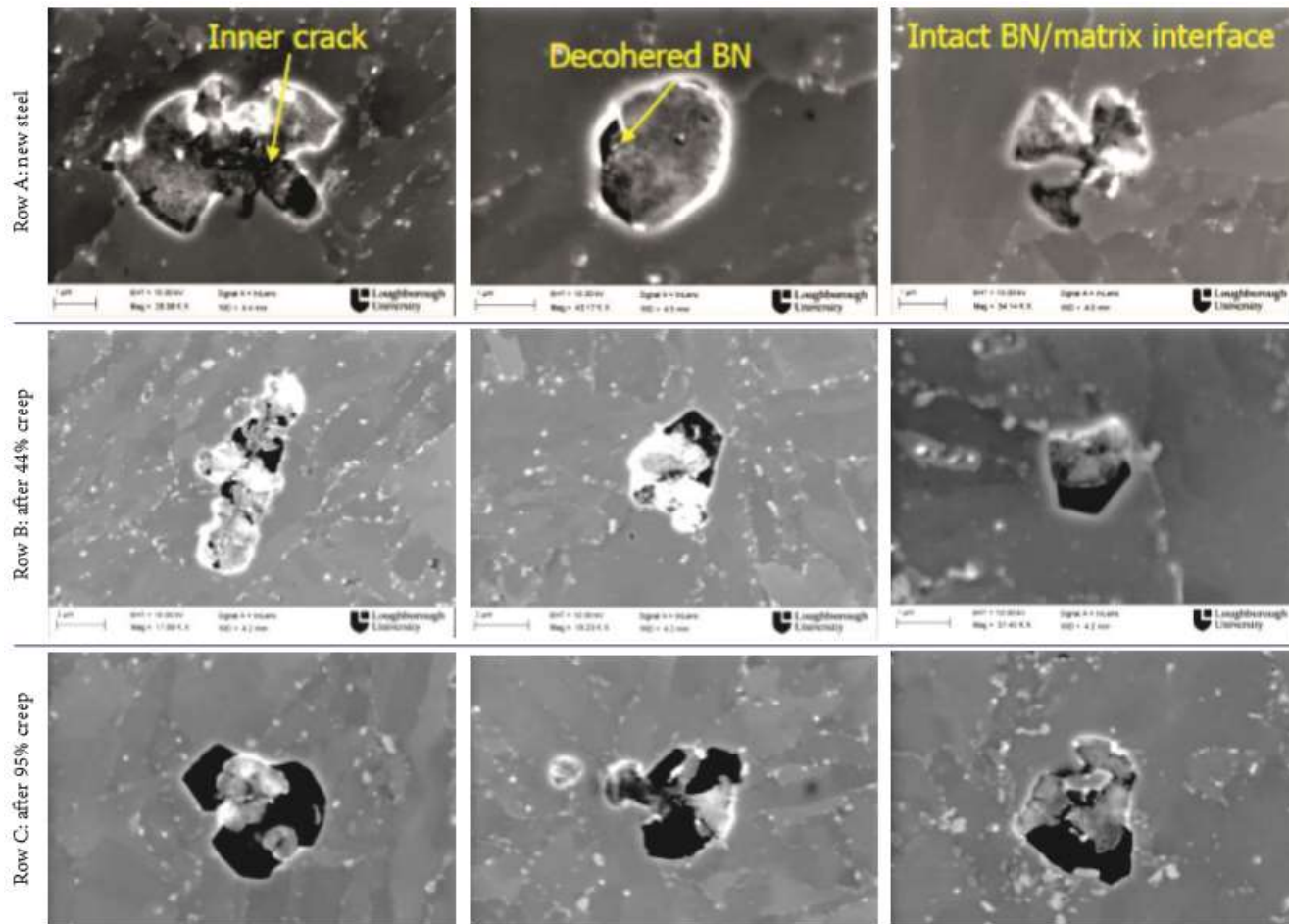
Original EBSD image



200 μ m

Reconstructed prior
austenite image

Cavity formation in Grade 92 steel



Parker & Siefert in
Advances in Materials
Science and Engineering
2018; ID 6789563

Future Work and Summary

Summary

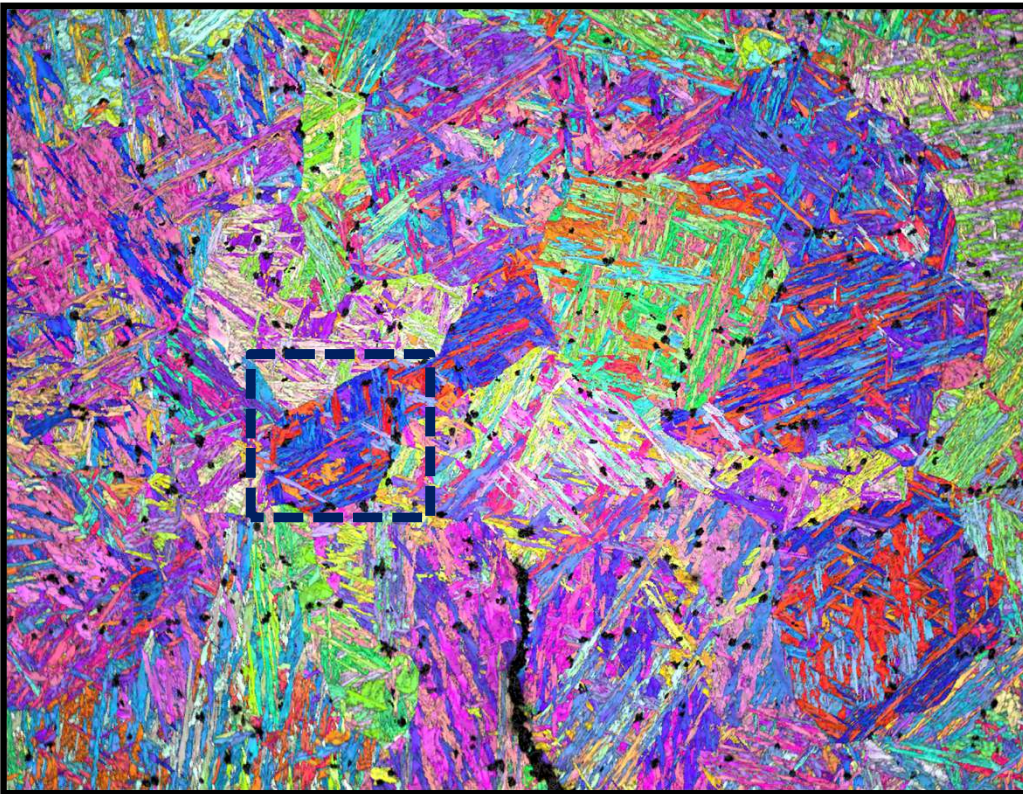
- Phase I – evaluate the influence of renormalization
 - Where BN is present, normalization $\geq \sim 1150^{\circ}\text{C}$ and accelerated cooling can put the nitrogen and boron back into solution
 - However, damage susceptibility cannot be improved if other inclusions are present in sufficiently high quantities
 - Ca- and Al-rich have a stability $>$ melting
 - MnS has a stability $\sim \geq 1400^{\circ}\text{C}$
- The influence of the renormalization is not having a greater benefit in multiaxial tests and simulated HAZ tests because we have not been able to fully remove the nucleation sites for damage which are shown to be Ca-rich
 - Microstructure assessment being finalized for analysis of BN

The influence of damage governs behavior in the long-term for 9%Cr martensitic steels

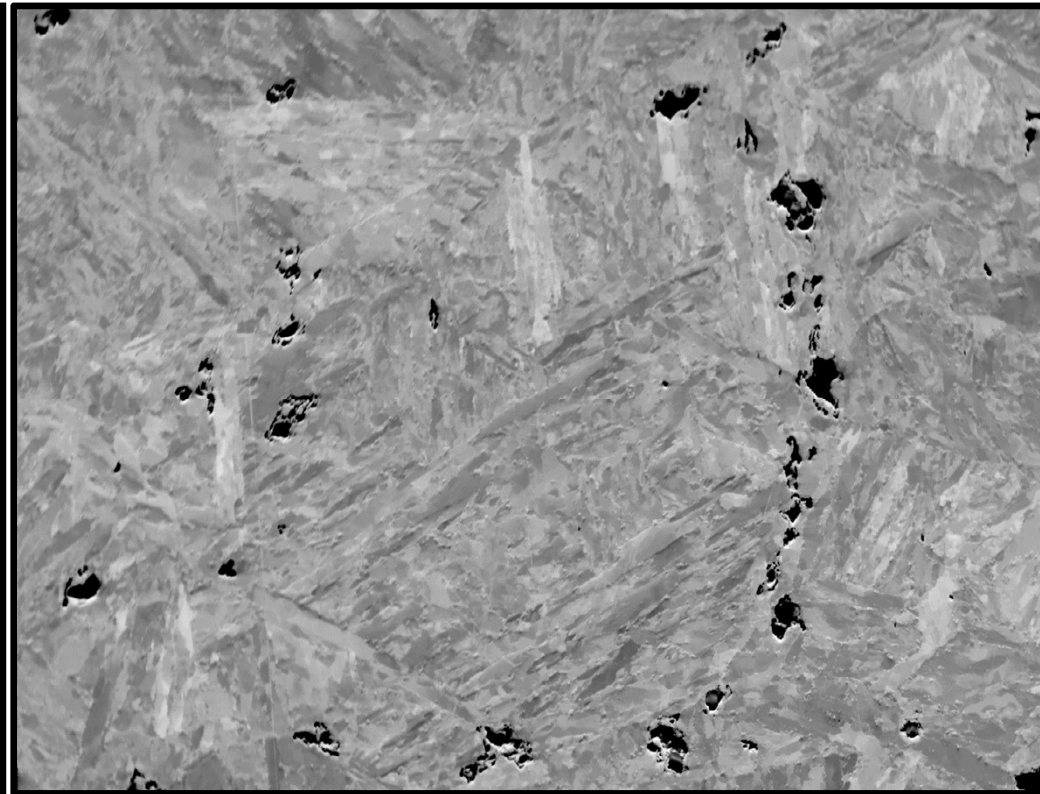


Together...Shaping the Future of Electricity

Localized EBSD at Prior Austenite Grain



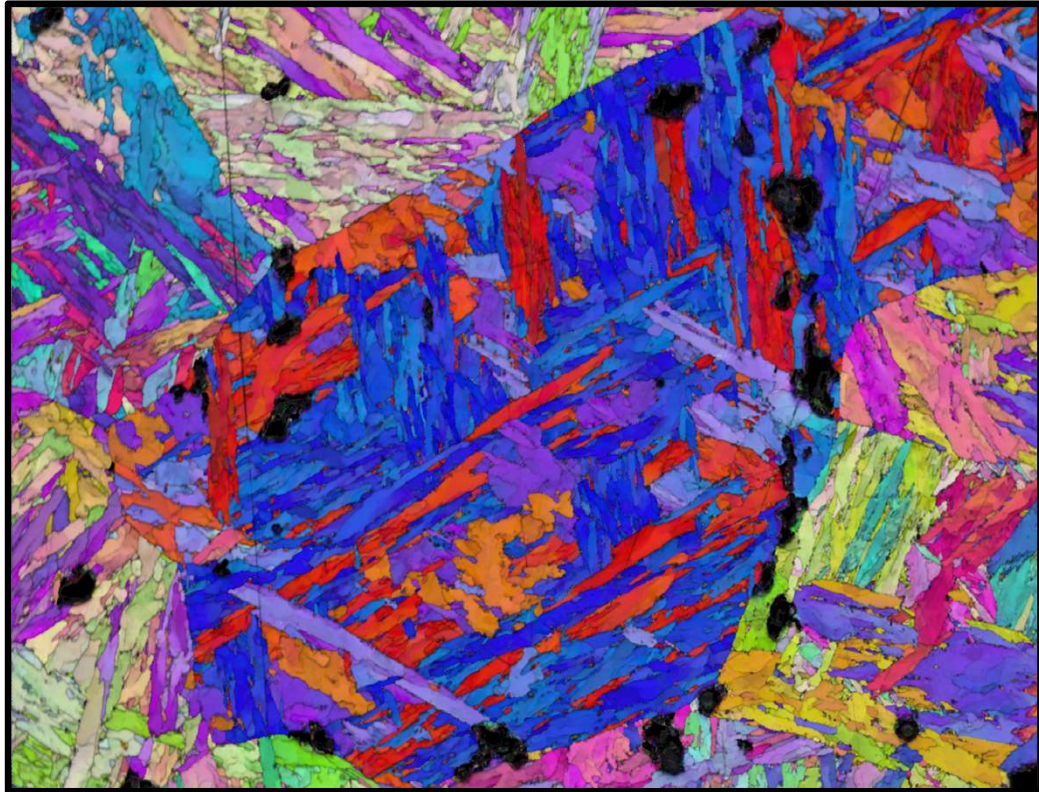
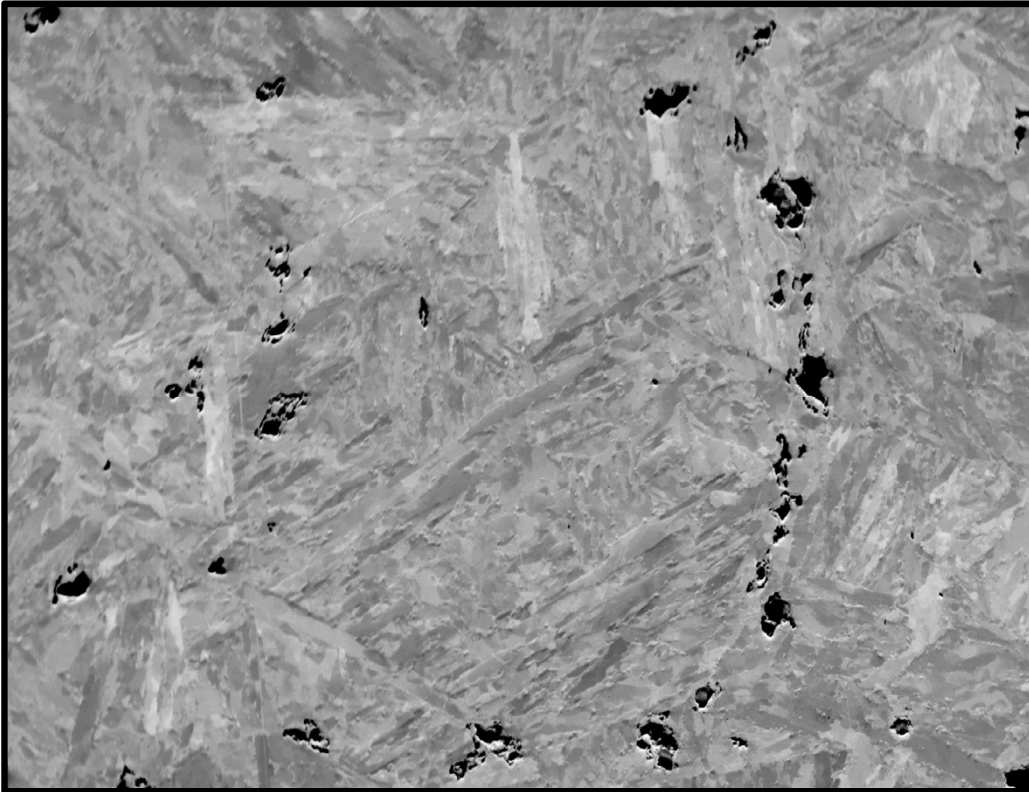
200 μm



50 μm

Inverse Pole Figure Image from EBSD Scan

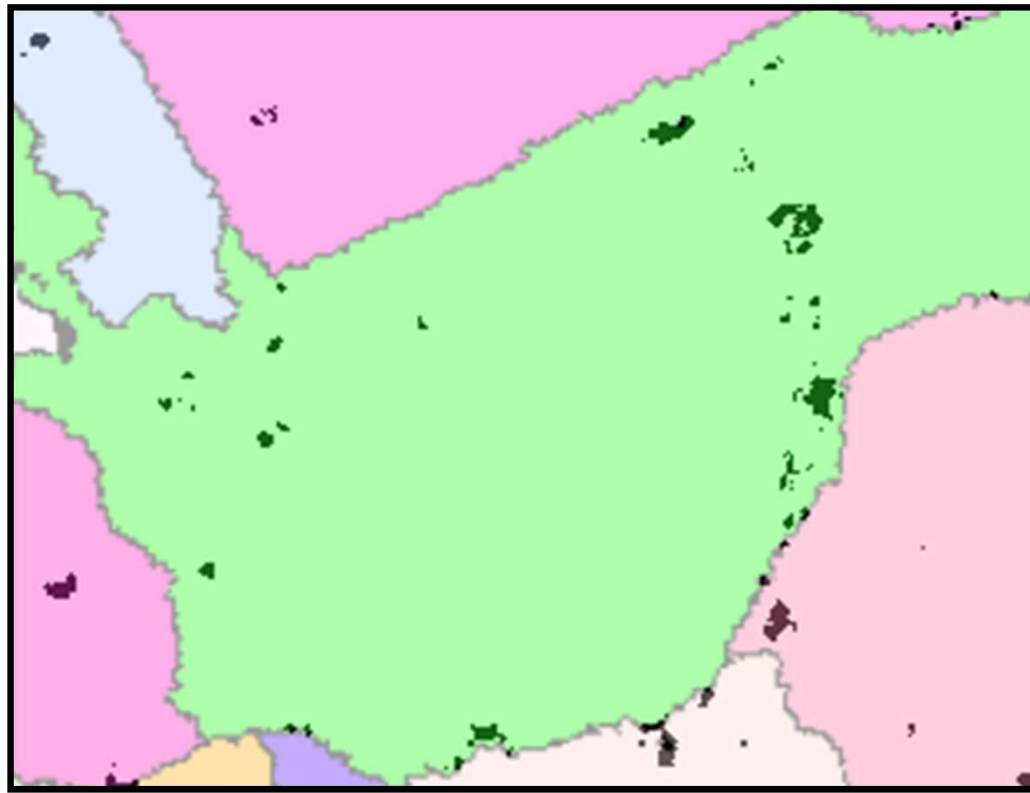
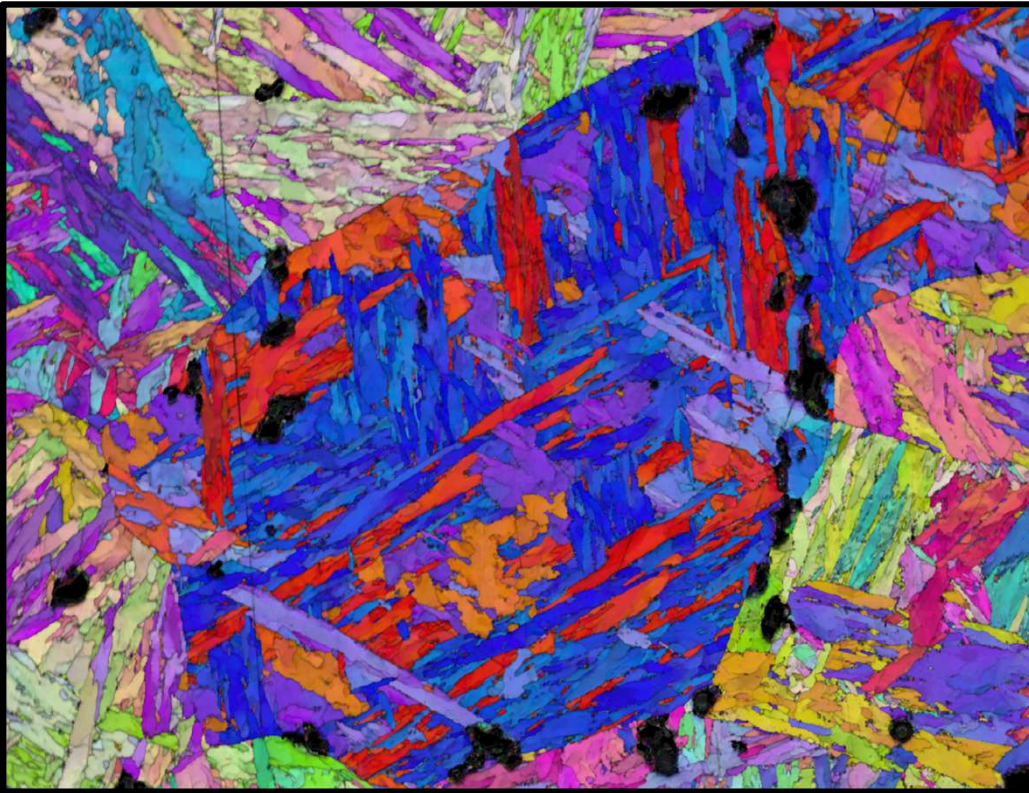
Damage appears to be predominately at prior austenite GB. However.....



50 μm

Inverse Pole Figure Image from EBSD Scan

..it is not really true. Major portion of damage appears to be within Austenite Gb



50 μm