

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

DE-FE002620



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**Crosscutting Research and Rare Earth Elements Portfolios Review**  
April 21, 2016

# Setting the Stage for World Power Market

## ■ USA

- There is immense commercial pressure to increase efficiency in state-of-the-art combined cycle gas turbine (CCGT) plants
- Within the immediate future, steam outlet temperatures in heat recovery steam generators (HRSGs) will achieve  $>600^{\circ}\text{C}$

## ■ Asia

- There is immense commercial pressure to increase efficiency in coal fired power plants (i.e. today's ultra-supercritical power plants are operating with steam outlet temperatures approaching  $625^{\circ}\text{C}$ )
- Steam outlet temperatures in advanced ultra-supercritical power plants are being planned within the range of  $700$  to  $760^{\circ}\text{C}$

**Regardless of end-use application there is an increasing need for materials with optimized thermal/creep properties and with a minimized cost impact → CSEF steels**

# **Background, A Summary of >5 Years of EPRI Research in Grade 92 Steel**

# Grade 92 → 9CrWNbVNB CSEF Steel

## Not all Grade 92 is the same

Conventionally Measured Elements

Gr92 Heat	Co	Cr	Ni	P	Mn	Mo	Si
BM A	0.015	8.797	0.38	0.009	0.49	0.43	0.211
BM B	0.016	8.776	0.25	0.012	0.54	0.33	0.182
BM C	<0.001	8.939	0.19	0.009	0.40	0.43	0.252

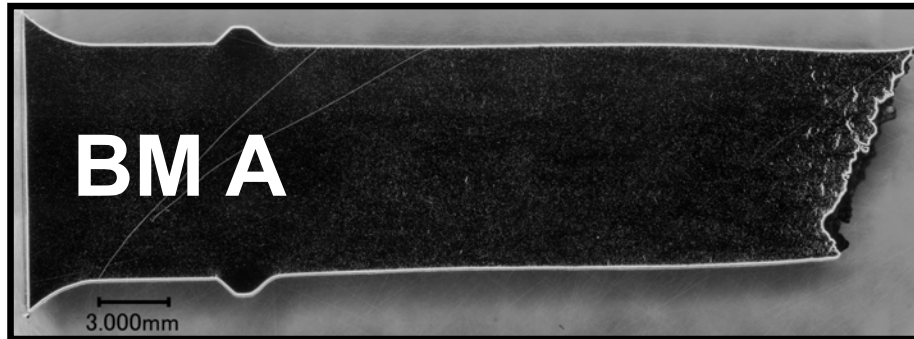
  

Gr92 Heat	B	C	N	Nb	V	W
BM A	0.0041	0.113	0.045	0.062	0.188	1.836
BM B	0.0041	0.131	0.0468	0.056	0.191	1.617
BM C	0.0042	0.093	0.0508	0.054	0.189	1.794

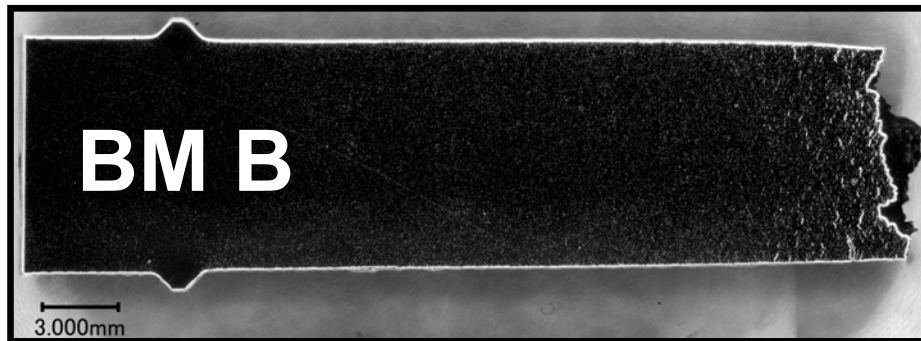
Gr92 Heat	Al	As	Cu	O	S	Sb	Sn
BM A	0.002	0.0064	0.189	0.0053	0.008	0.0016	0.016
BM B	0.015	0.0082	0.135	0.0022	0.001	0.001	0.008
BM C	0.001	<0.0001	0.001	0.0043	0.001	<0.0001	<0.001

**Also analyzed, with no detected amount: Pb, Bi, La, Nd, Ta, Ti, Zr and Ca ≤0.002**

## The Trends in Controlled Composition is Observed in Grade 92 [650°C/90 MPa]



- 3,945 hours
- 26.0% ROA



- 8,593 hours
- 16.3% ROA

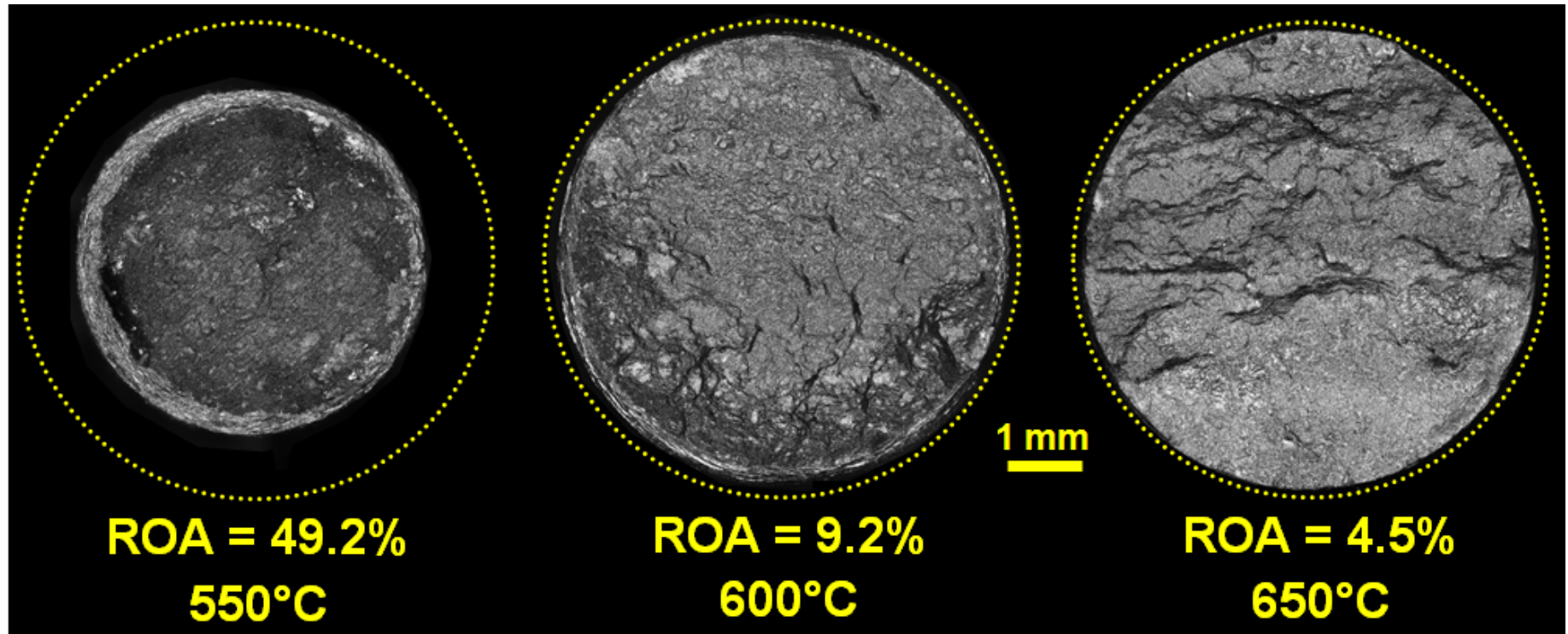


- 14,190 hours
- 42.4% ROA

**3.6X increase in strength;  
Observable increase in ductility**

# Comparison of Creep Ductility for a Damage Susceptible Heat of Grade 92

Time to Rupture for all Specimens ~26k hours



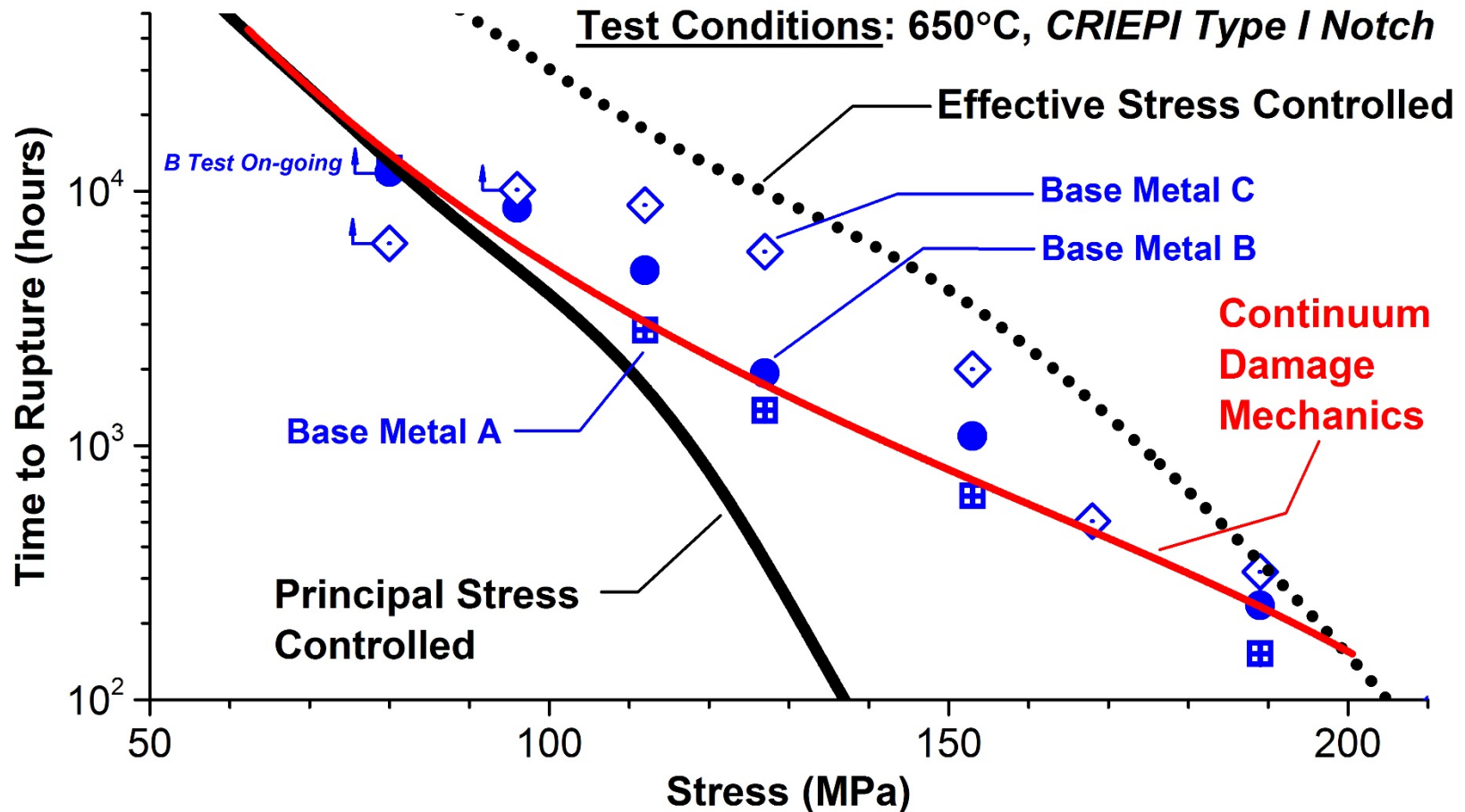
State-of-the-art designs will operate in temperature regimes  $\geq 600^{\circ}\text{C}$

# Recent EPRI Database Development

- EPRI has evaluated performance of Grade 92 steel for three unique base materials (the previously mentioned “BM A”, “BM B” and “BM C”). Testing included
  - Uniaxial creep
  - **Notch bar creep (two notch designs), and interrupted**
  - Creep-fatigue to ASTM E2714-09 with various hold times, strain rates, test temperatures, etc.
  - Cyclic stress relaxation
  - Fatigue
  - Cross-weld tests including feature tests in selected heats
- >500k hours of testing and ~\$3 million in leverage from industry and collaborative partners



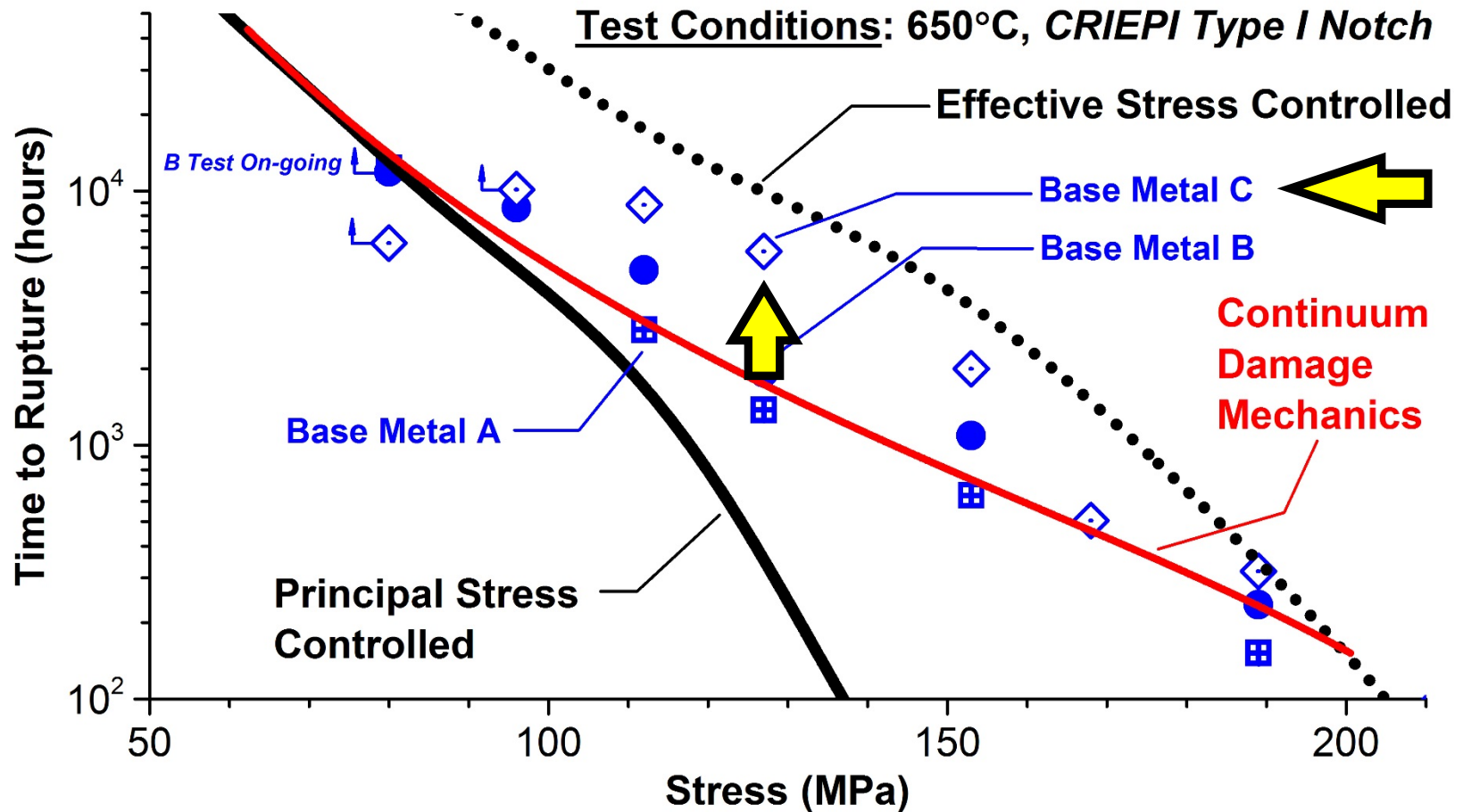
# Research has Shown the Value in Notch Bar Creep Testing as a Feature Test which is Relevant to the Development of Damage in Power Plant Components



**There is a clear transition to principal stress controlled damage (i.e. initiation and linking of creep cavities)**



# Research has Shown the Value in Notch Bar Creep Testing as a Feature Test which is Relevant to the Development of Damage in Power Plant Components



An upper-bound strength, super-clean heat (BM C) shows a distinct offset to principal stress controlled cavitation damage

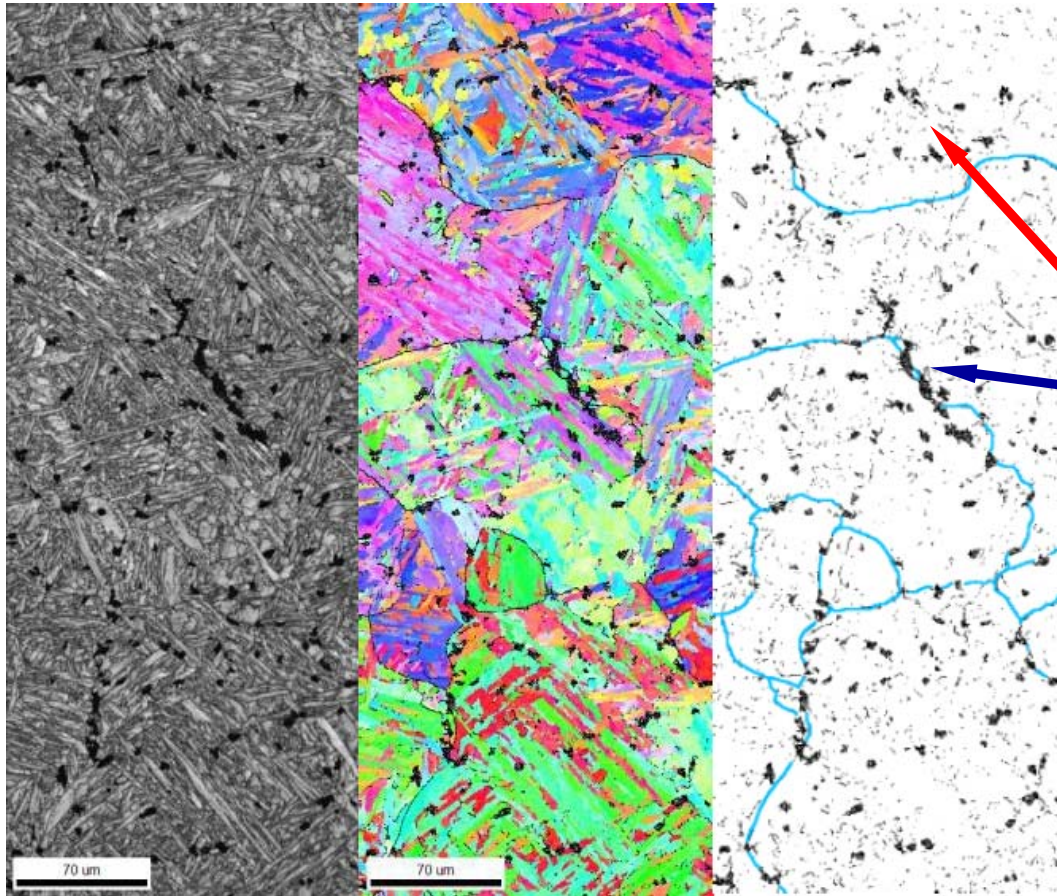
# The Concern Regarding Low Ductility

- Traditionally in material development for the power industry, creep ductility (i.e. resistance to damage) has been given little consideration
- However, the risk to catastrophic failure due to low damage tolerance is a reality where
  - Mechanical notches exist due to poor design considerations
  - Metallurgical notches exist to fabrication (i.e. weldments)
- The realities regarding creep ductility, even when investigated, are often improperly identified:
  - Uniaxial creep versus notch tests
  - “Small sample” cross-weld versus feature cross-weld tests

**Where CSEF steels possess high susceptibility to damage, these grades or heats trend to NOTCH WEAKENING behavior where proper testing produces relevant results**

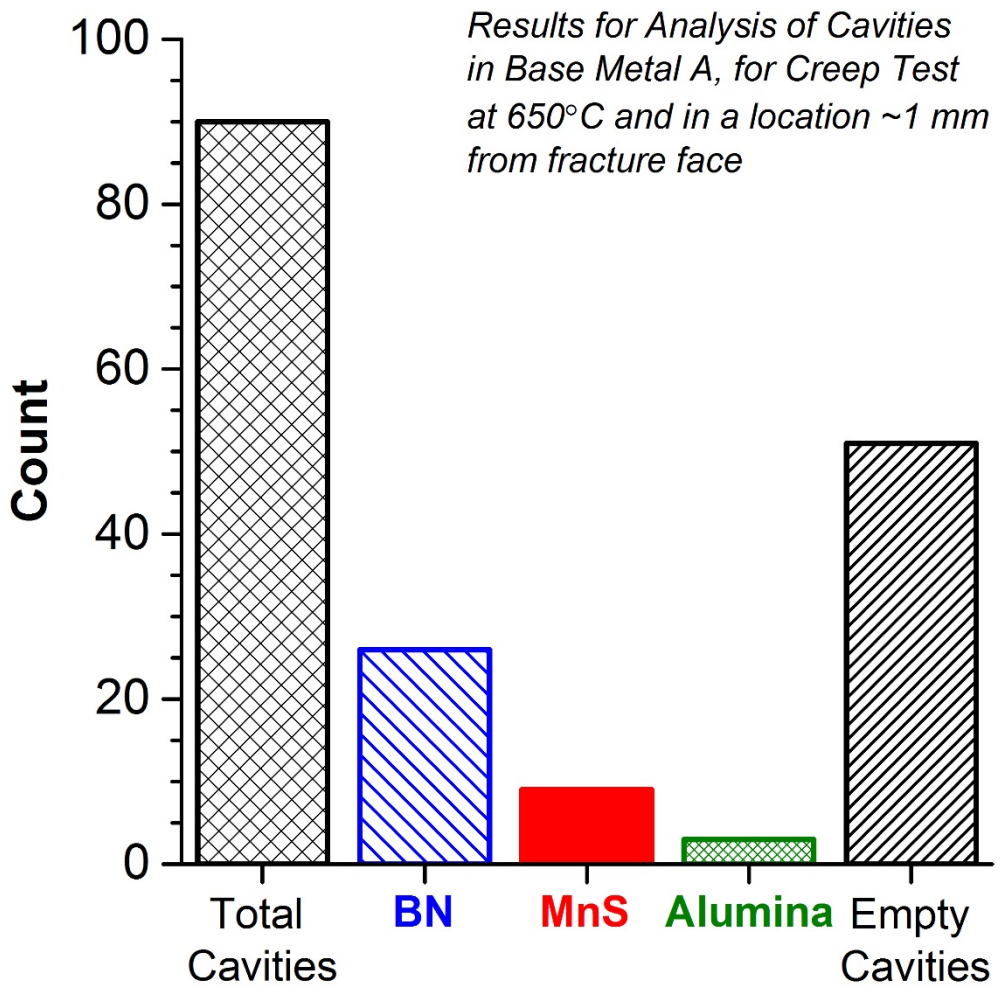
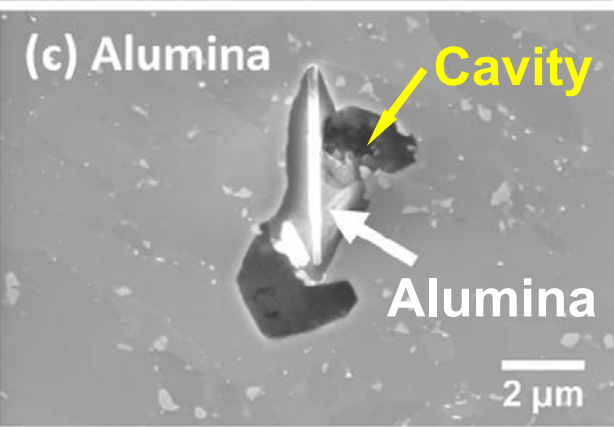
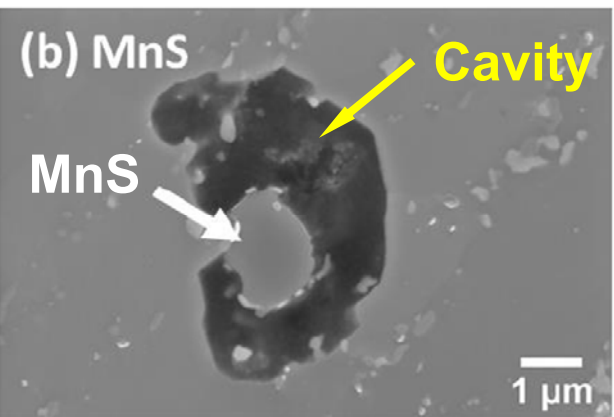
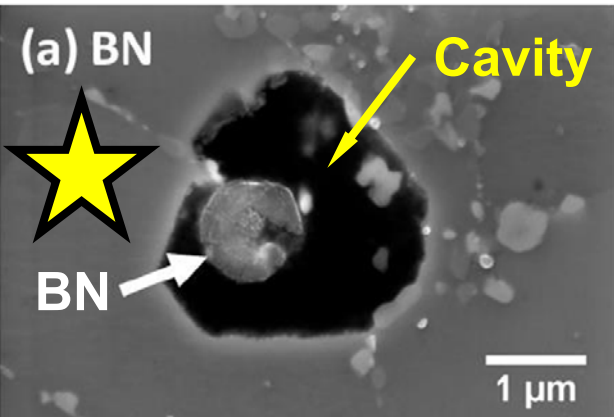
**The macro-level assessment is critical to validating models to apply to in-plant components. Since the model predicts a trend to cavitation-dominated damage, it is important to evaluate damage from a micro-level using advanced electron optics and confirm this observation**

# Micro-level Evaluation of Grade 92 Base Material Samples indicates that Damage is NOT Isolated to Prior Austenite Grain Boundaries in these Steels

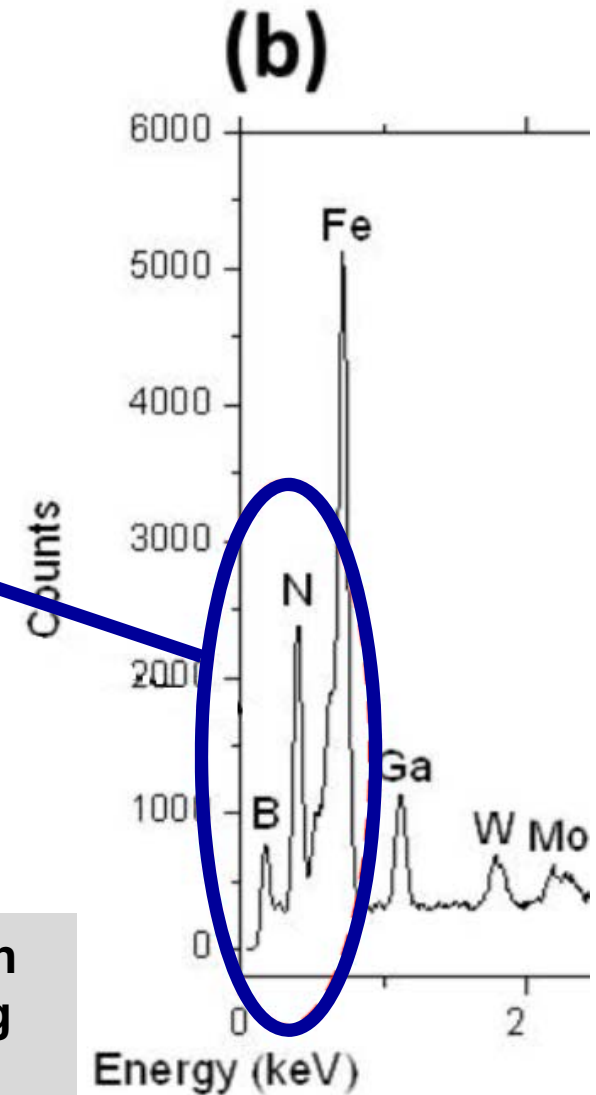
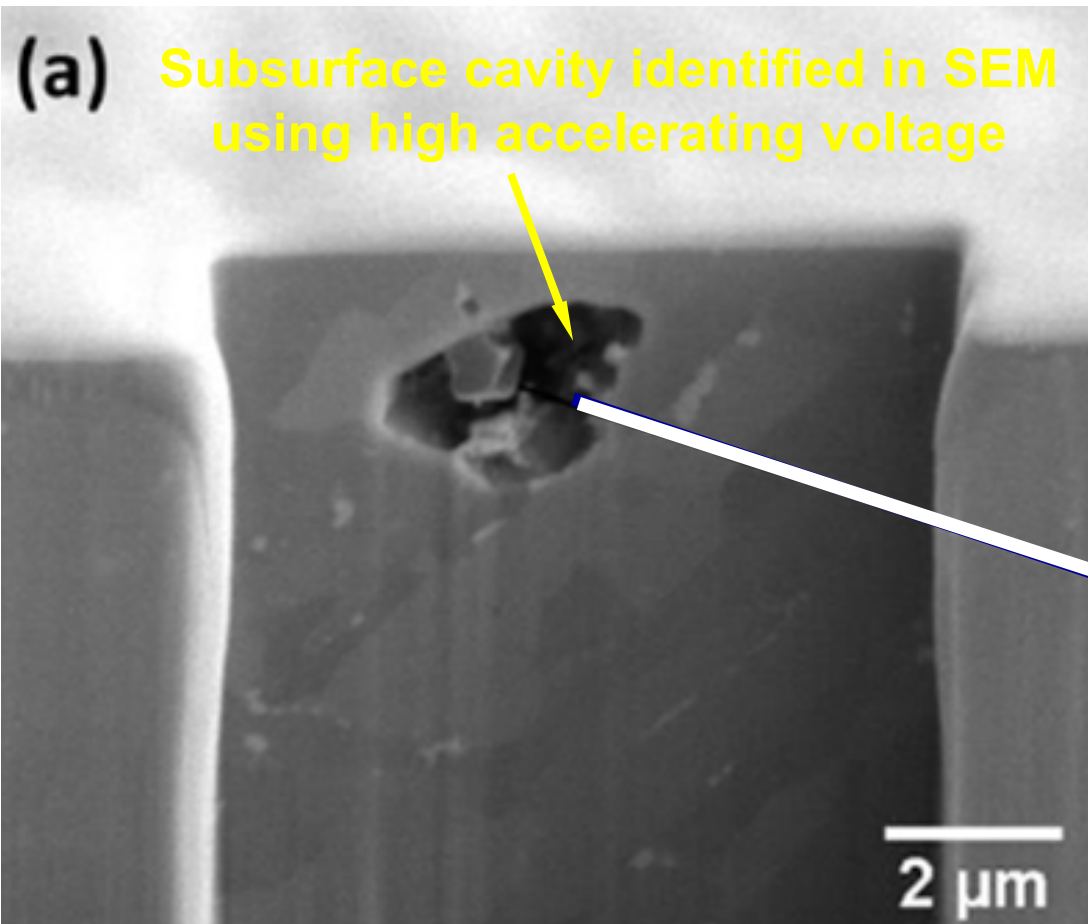


- Damage must be forming in or on multiple features since damage is clearly **within the grain** and on **grain boundary**
- This contradicts much of the literature suggesting:
  - Poor characterization in most assessments
  - Potential for multiple damage modes where the reported results *are from a pedigreed researcher*

# Micro-level Evaluation of Grade 92 Base Metal Damage shows a Strong Correlation with Boron-nitride (BN) and a Weaker Correlation with MnS and Alumina

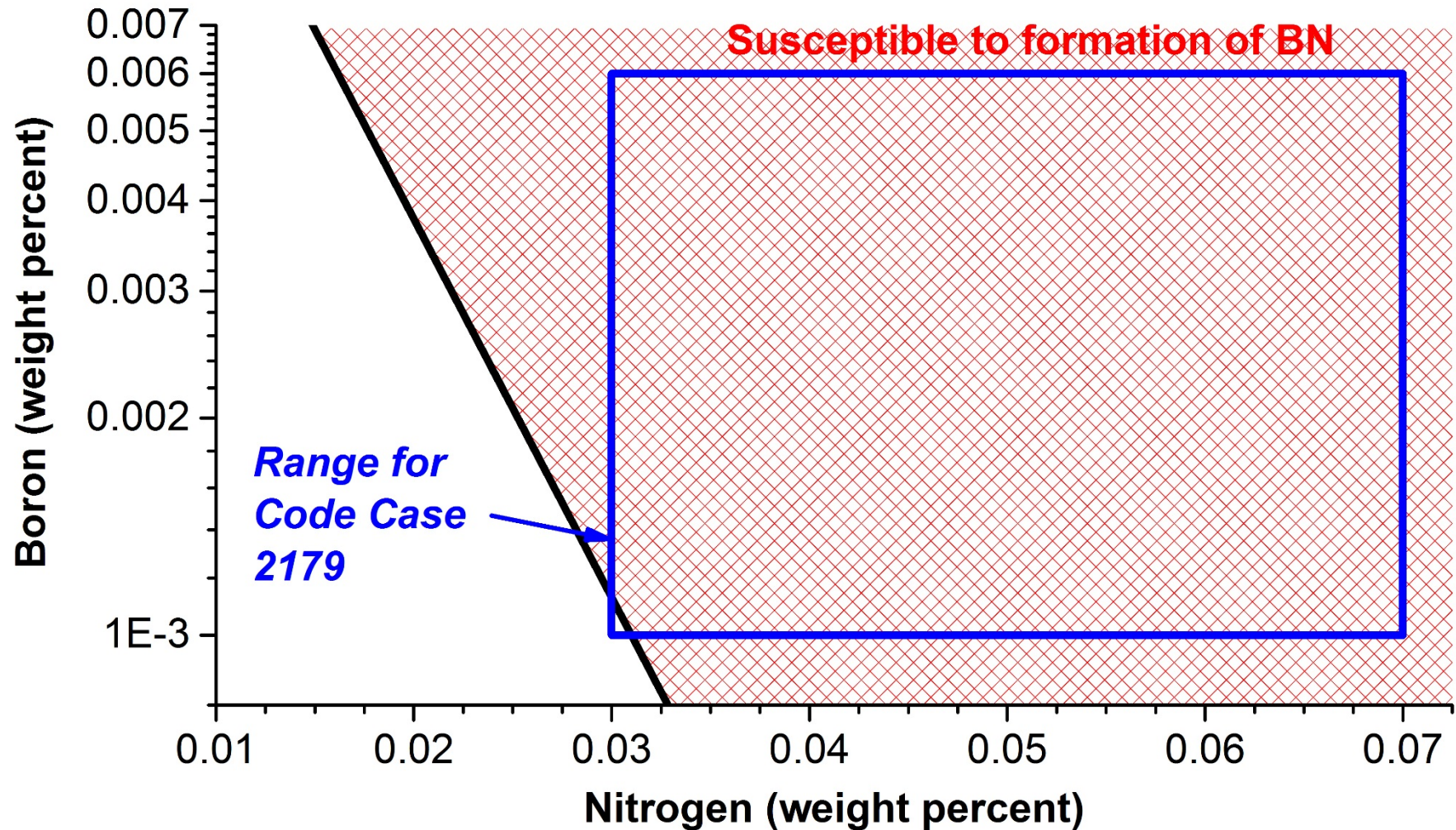


# Confirmation of Cavitation was Performed for Each Evaluated Heat of Grade 92 Steel



To confirm the link to BN, sub-surface cavities such as in (a) were removed by focused-ion beam milling BN is confirmed in (b) by EDS

# All Heats of Grade 92 are Susceptible to the formation of BN (hence the development of MARBN-type steels such as SAVE 12 AD and others) – From Abe Diagram



# Quick Summary

- Eliminating the presence of BN in Grade 92 steel is important to reduce the “easy” nucleation sites and delay the transition to principal stress controlled damage
  - Accomplished by an unconventional, **high temperature normalization ~1150°C**
  - And “sufficiently fast” cooling rate to avoid BN reformation
- Optimizing an existing, Code-approved material is desirable from a practical and database standpoint
  - Currently, **Grade 92** is the highest strength approved CSEF steel in ASME Code (Code Case 2179)
  - Material is no longer exclusive to a single manufacturer and the supply chain already exists for manufacturing the needed components:
    - Castings
    - Fittings
    - Plate
    - Pipe
    - Tube

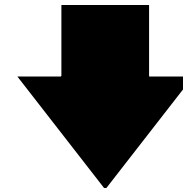


# On the Reduction/Removal of BN from the Matrix

- All of the evaluated Grade 92 steels show damage associated with BN
- Dissolution of BN is accomplished through a sufficient peak temperature (see table on right)
- Preventing the re-formation of BN is believed to be a cooling rate dependent issue (i.e. may need to impose water-, oil- or accelerated air-cooling)
- Additionally, an “unconventionally” high normalization and cooling can lead to optimized precipitate structure

Temperature (Time = 2 hours for all conditions)	Result <sup>1</sup>
1200	BN Fully Dissolved
1175	
1150	
1125	
1100	Not Dissolved

<sup>1</sup>Simulated experiments for BMA using a Gleeble Thermomechanical Simulator

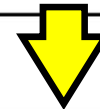


***For Grade 92, proper normalization needs to be conducted at 1125°C minimum and 1150°C target***

# An Interesting and Relevant Point on Normalization from Code Case Data Package for SAVE 12AD

**Note: SAVE 12AD is a 9Cr-3W-3Co-Nd-B (Nb, V, N)**

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

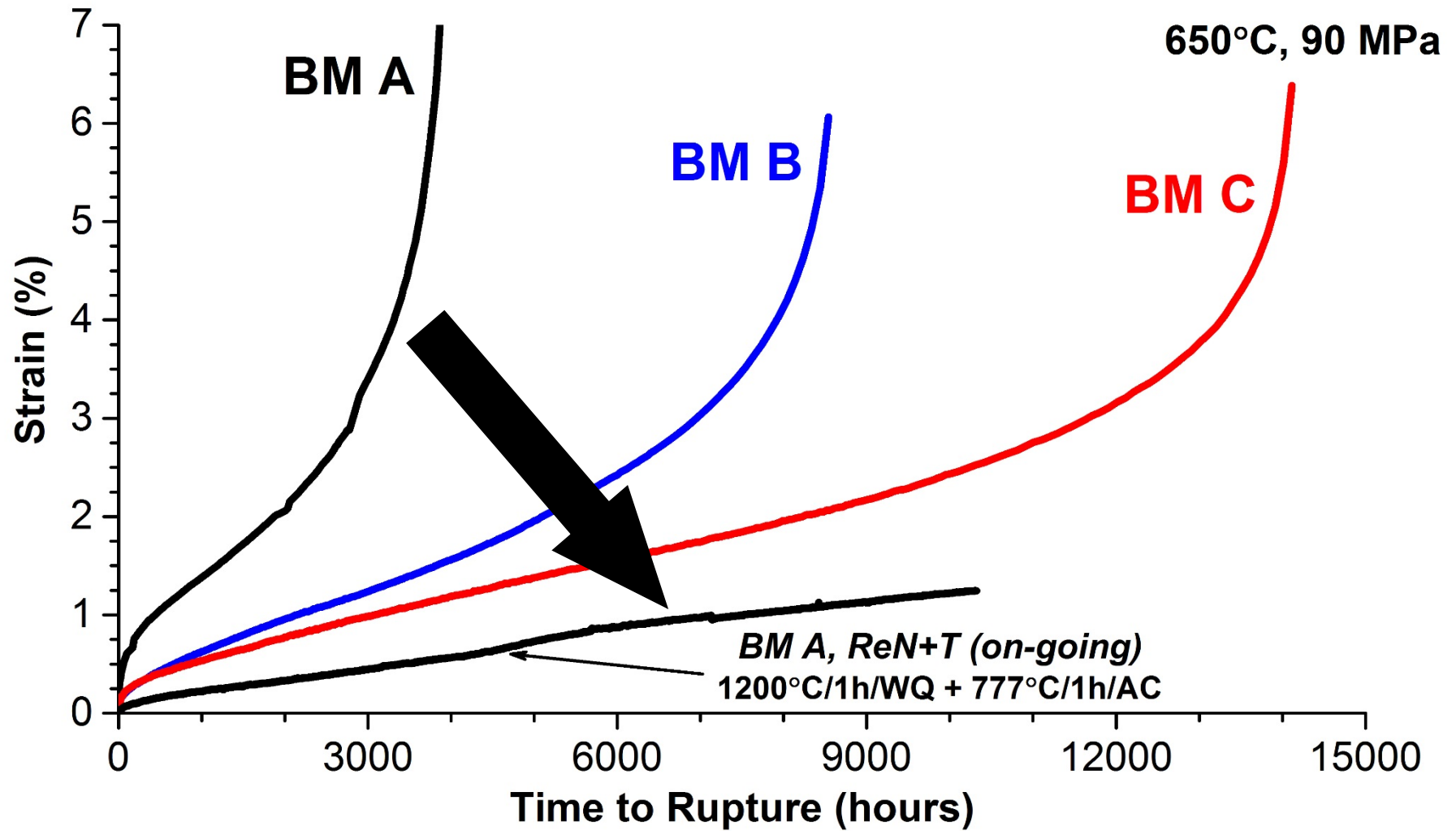


# Grade 92 Uniaxial Creep Specimens

Specimen	Base Metal	Test Conditions		Life (hrs)	Elong. (%)	ROA (%)
		Temp. [°C, (°F)]	Stress [MPa, (ksi)]			
G92-33	BM A	650 (1202)	90 (13.05)	3,945	11.2	26.0
G92M-21	BM B			8,593	8.1	16.3
G92J-23	BM C			14,190	11.2	42.4
10766-1CRP	BM A (ReN+T) <sup>1</sup>			<b>&gt;14,190 (on-going)</b>		

- As a simple experiment, the poorest performing material (in terms of strength and ductility, BM A) was re-normalized at 1200°C/1h/WQ and tempered (777°C/1h/AC)
- To date, this material exceeds performance [strength] as compared to BM C. Ductility/damage will be confirmed upon failure

# Comparison of Grade 92 Uniaxial Creep Specimens

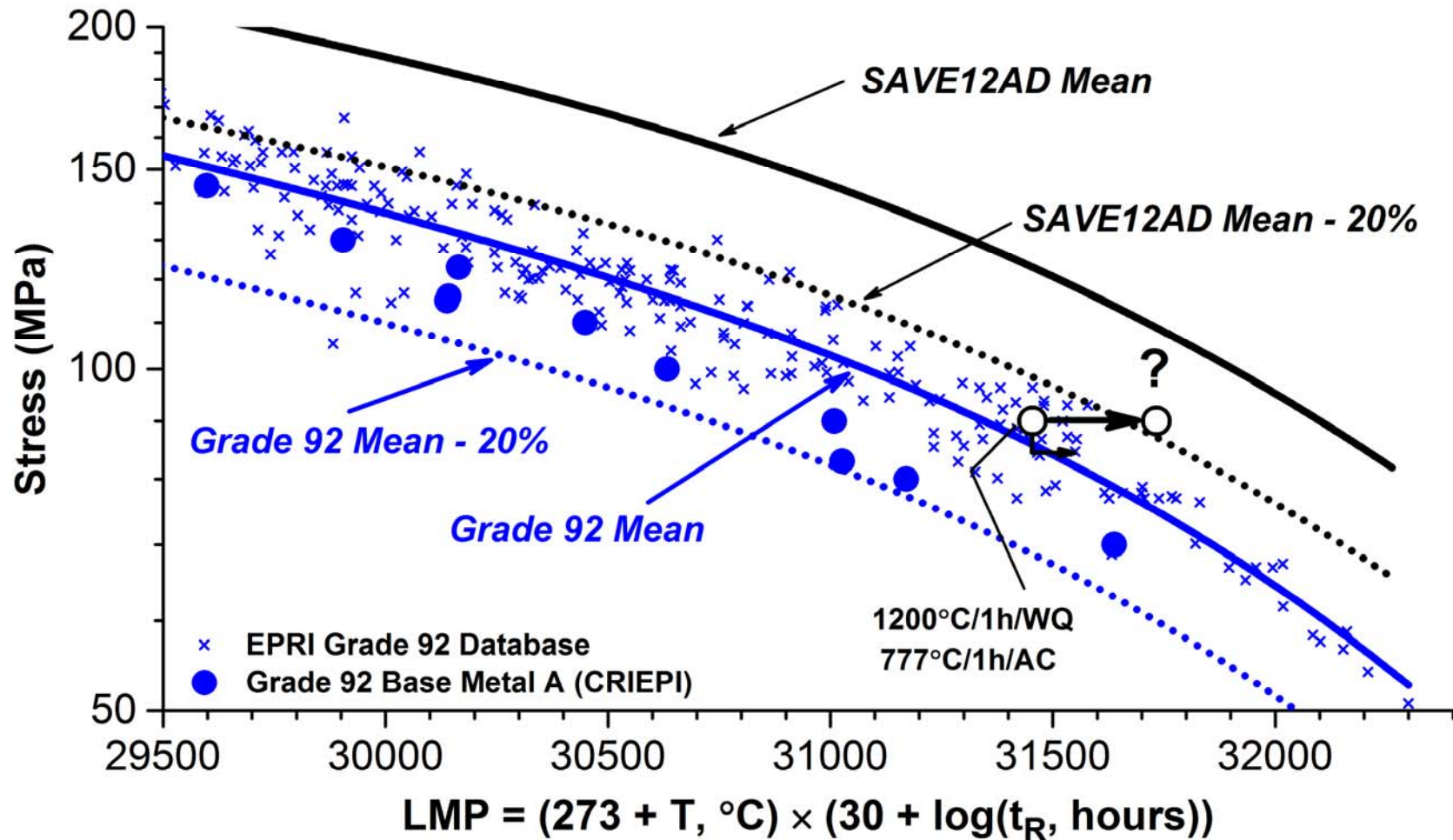


**Based on this information and promising approaches for modeling and material heat treatment optimization, EPRI was awarded a project sponsored by DOE**

# Motivation for EPRI Project

- Increase the resistance to damage [i.e. creep ductility]
  - We can potentially increase the creep ductility in Grade 92 steel by:
    - Reducing the void nucleation sites (i.e. remove BN)
    - Delay the transition to principal stress controlled damage (i.e. very clean composition)
- Increase the deformation resistance [i.e. creep strength]
  - We can potentially increase strength in Grade 92 steel by:
    - Optimizing the type of precipitate
    - Optimizing the precipitate composition
- **Phase I – optimized heat treatment of a commercial heat of Grade 92**
- Phase II – optimized heat treatment of a “super clean” commercial heat of Grade 92

If both the Composition and Processing are Optimized for Grade 92, we may reasonably Expect Performance within the Scatter-band for SAVE12AD



Heat Treatment may also be effective in delaying the transition to principal stress controlled damage

# Tasks

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



## Task 2.0 – P92 Alloy Procurement and Processing

- Donated pipe measures 20” OD X 5.27” WT X 4 feet long
- One section left in the as-manufactured state
  - Normalization = 1065C for 2.75 hours + Fan Cool
  - Tempering = 775°C for 5.5 hours
- A second section given an optimized heat treatment
  - Normalization = 1125°C (2055°F) for 2 hours minimum (Target 1150°C) + Oil Quench
  - Tempering = 775°C (1425°F) for 5 hours

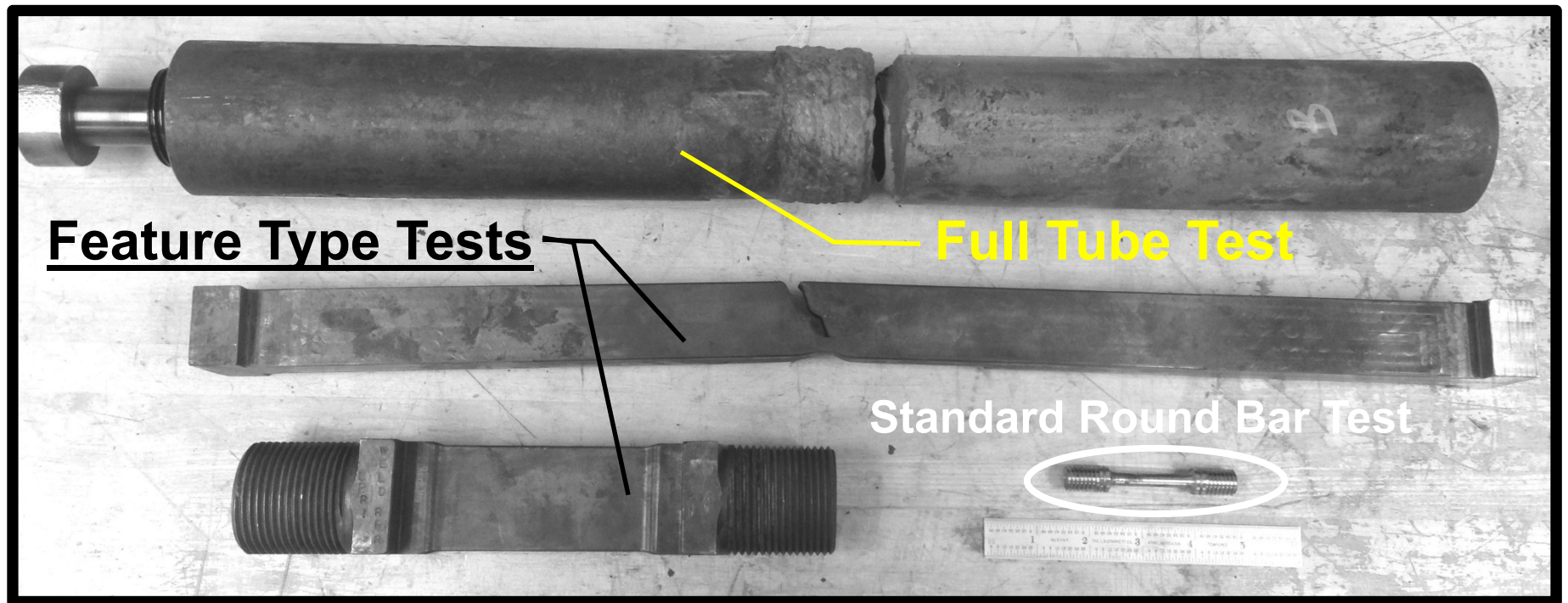
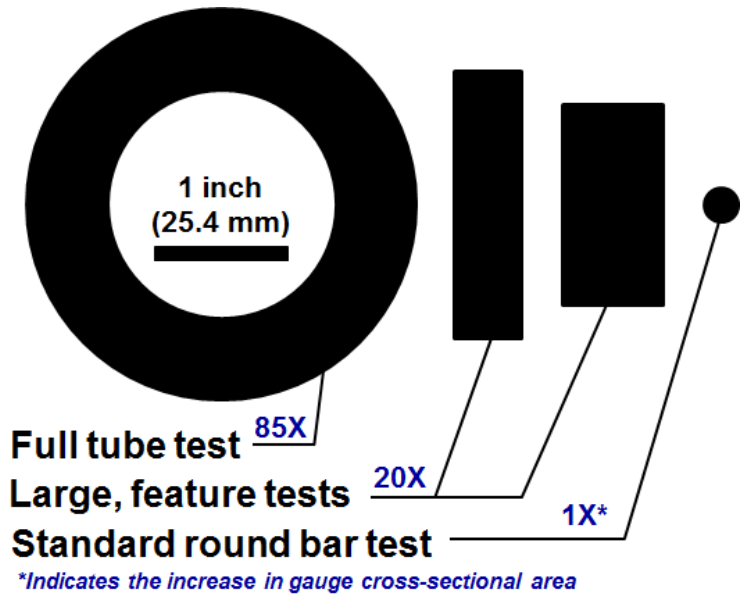
C	Mn	P	S	Si	Ni	Cr	Mo	V	Ti
0.084	0.47	0.008	0.0013	0.238	0.17	8.693	0.43	0.192	<0.002
Co	W	Nb	B	N	Al	As	Cu	Sb	Sn
0.014	1.86	0.064	0.0023	0.0480	0.002	0.004	0.152	0.0012	0.007

***Also analyzed, with no detected amount: Pb, Bi, La, Ta, Zr and Ca ≤0.002***

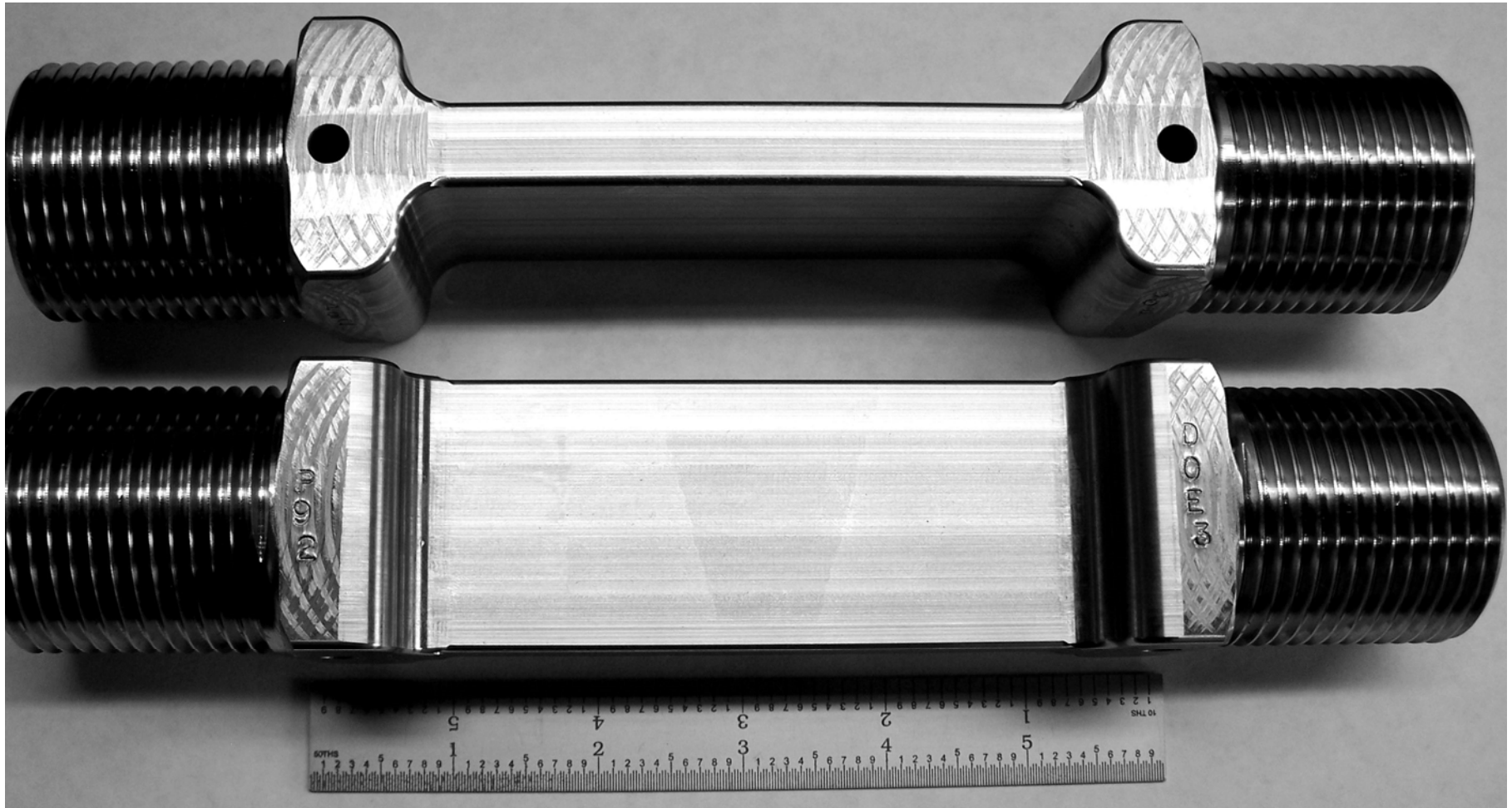
# Task 4.0 – Planned Testing is Underway

- Plain bar creep tests on optimized Grade 92
- Notch bar creep tests on optimized Grade 92
- Sequential Fatigue-Creep tests
  - Low cycle fatigue + Creep
  - Creep + Tensile
- Simulated HAZ plain bar creep tests on conventional and optimized Grade 92
- Feature Cross-weld Creep tests
- [ORNL Task 7.0] Pressurized and pressurized + end load tests

# Comparison of Feature, Cross-weld Tests for Task 4.5 to Conventional Uniaxial Round Bar Sample



# Cross-weld Creep Feature Test Samples



- Weldment tests made in the oil quenched produced pipe
- Single vee weld (30° included angle) SMAW process + Grade 92-type filler metal and subcritical PWHT at 750°C/2h

# Task 5.0 – Microstructural Evaluation of Initial Material, Heat Treatments and as-Tested Samples

Sample	1150°C/1h/WQ	750°C/1h/AC	900°C/1m/AC	750°C/1h/AC
M1				
M2				
M3				
M4				
M5				
M6				
M7	As-received (1065°C/2.75h/FAC + 775°C/5.5h/AC)			
COMM-1	Optimized (1150°C/2h/OQ + 775°C/5h/AC)			

- Initial characterization to include:
  - Macro hardness measurements
  - PAGB and substructure
  - $M_{23}C_6$  and MX
  - BN dissolution
  - Cooling rate experiments
- Post-test characterization once samples begin to fail using similar methods for direct comparison

# Summary

- Resistance to deformation (i.e. creep strength) can be realized by an optimized heat treatment in 9Cr CSEF steels
  - It is not yet clear the role heat treatment will play in the resistance to damage, although the intent of the optimized heat treatment is to simultaneously modify both properties
  - In the minimum, it may be possible to delay the onset to principal stress controlled damage through heat treatment alone
- Demonstration of improved performance must be demonstrated by a combination of critical tests:
  - Notch bar creep tests
  - Feature tests in cross-weld creep
  - Multi-axial tube tests with pressure and end load
- The development of suitable CDM approaches for Grade 92 (conventional and optimized) are vital for potential Phase II component testing and design of components from optimized materials

# Looking into the Future (Potential Phase II Examinations)

- The performance of Grade 92 (and by extension Grade 91) can be further optimized through:
  - Careful control of composition (i.e. working towards a “superclean” composition)
  - Optimized tempering heat treatment (such as 750°C versus 775°C)
  - Potential for homogenization step (such as during pipe manufacturing)
- Ultimately, it may be possible to realize very high creep strength and acceptable creep ductility through existing materials as opposed to resource-extensive material property development for new alloy concepts

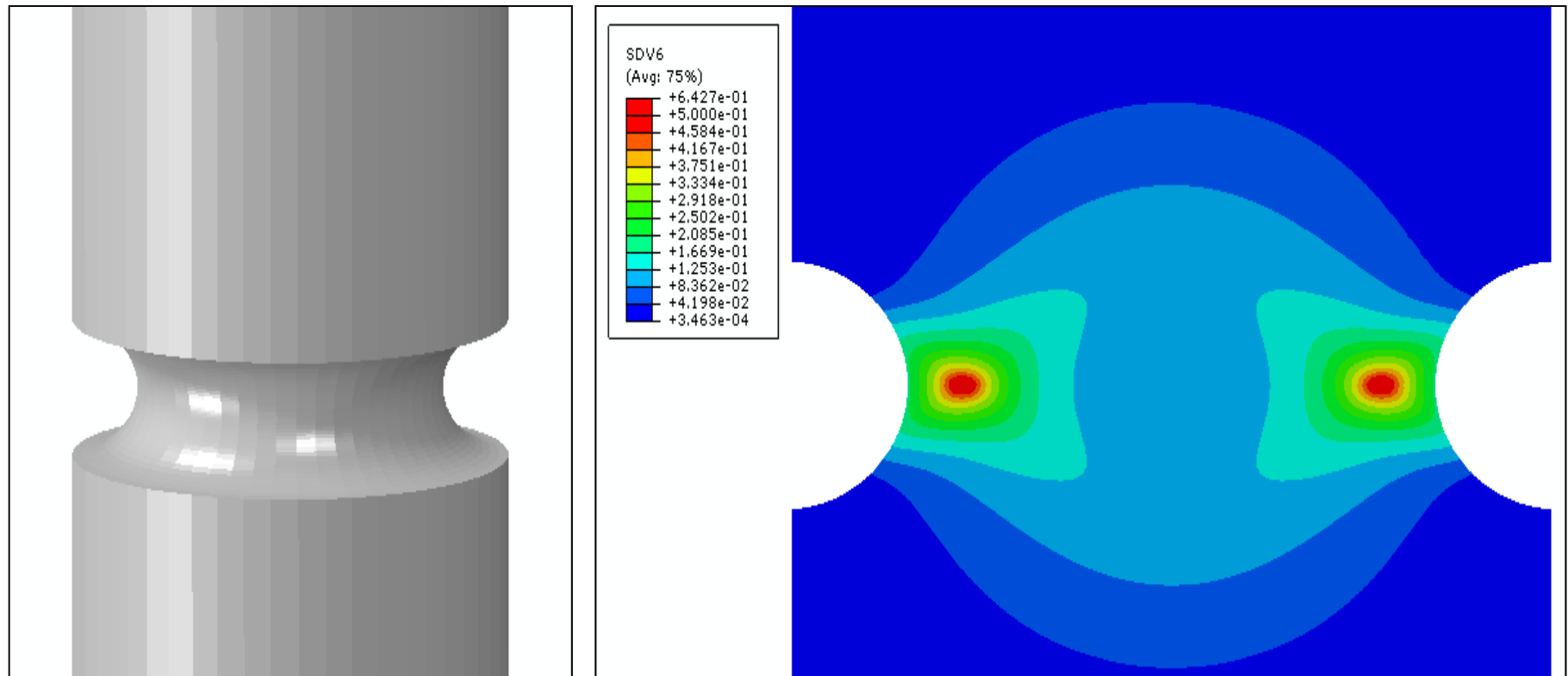
# Questions/Comments/Concerns





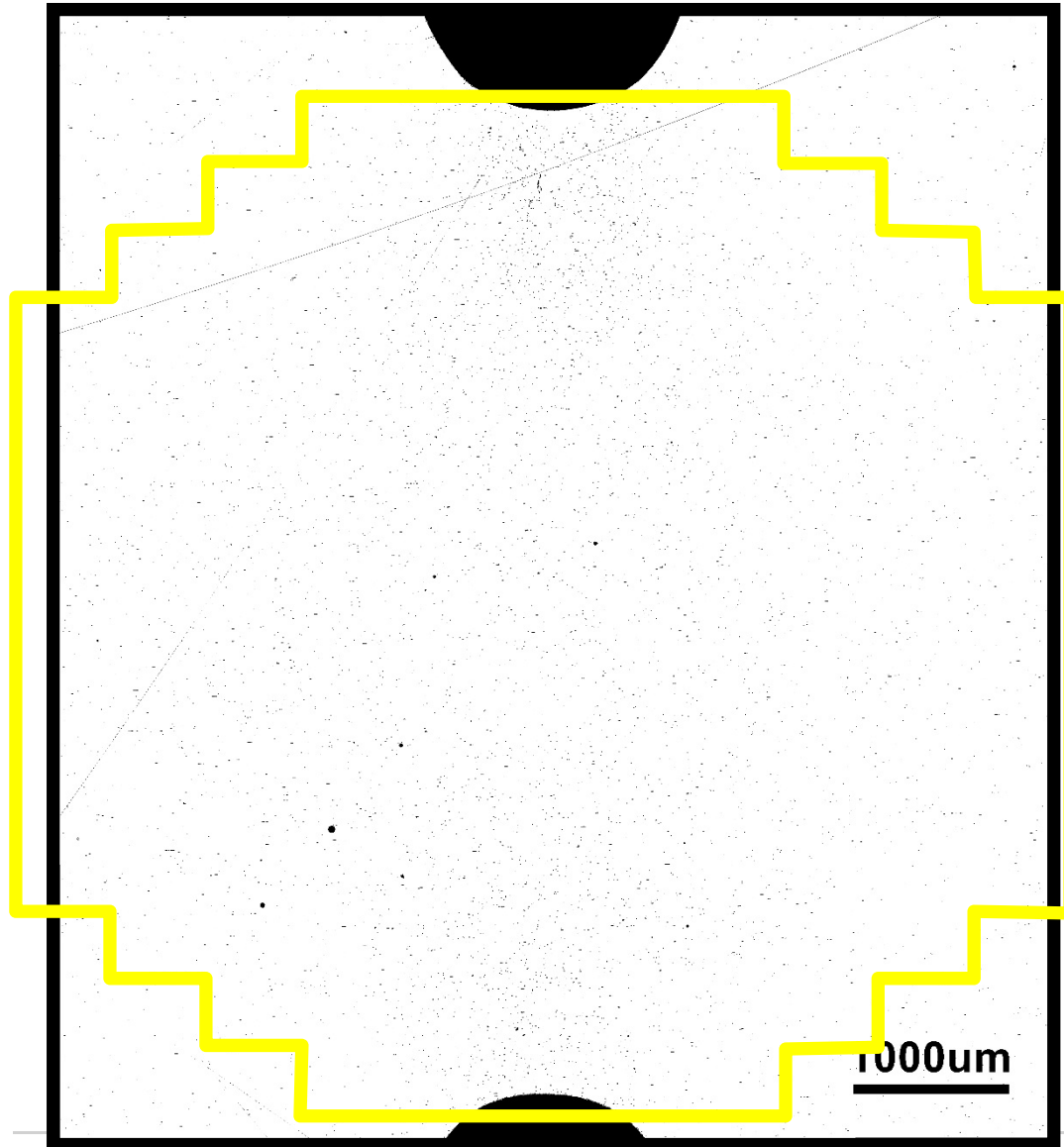
# Together...Shaping the Future of Electricity

# Tests under Uniaxial Creep, Notch Bar Creep, Creep-Fatigue, Cyclic Stress Relaxation have been Modeled to Exercise Developed Material Descriptions to Validate Continuum Damage Mechanics Approach



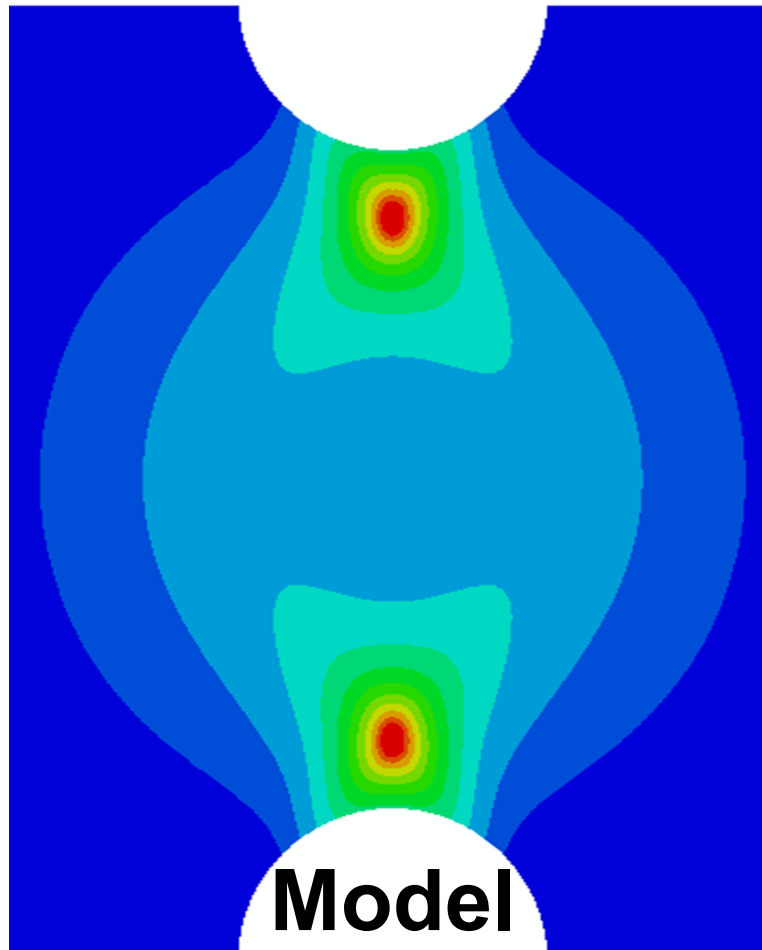
**Continuum Damage Mechanics Predicts Maximum Damage will be Subsurface**

# Detailed Macro-Assessment of Post-test Samples Includes Highly Accurate Laser Microscopy

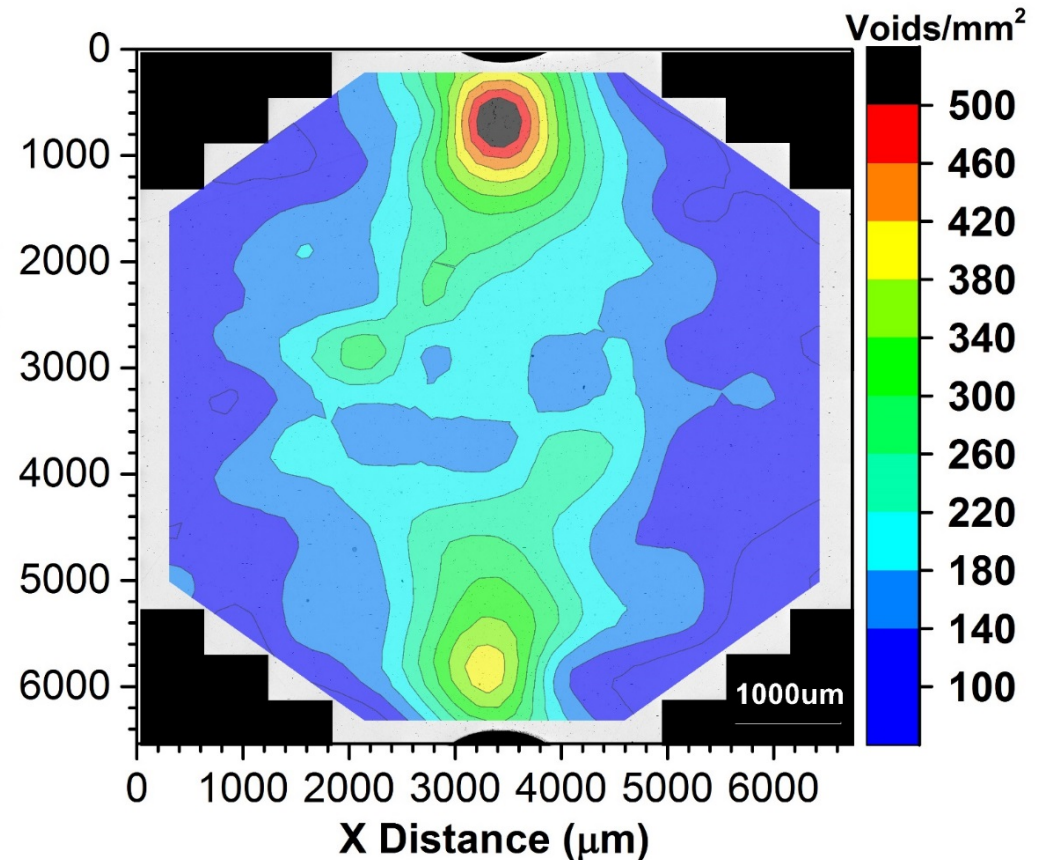


- Area highlighted by yellow was analyzed
  - 141 Total Images
  - 20X Objective (~400X magnification)
  - Void density calculated for each image and reported in voids/mm<sup>2</sup>
  - “Black area” for some images at notch was removed
- Data reported as:
  - “Heat Map” – Grid pattern
  - “Contour Map” Overlay onto the image

# Comparison of Model versus Actual Macro-Measurements shows Excellent Agreement



## Actual Measurements



**The Continuum Damage Mechanics Model can be Applied to Predictive, Component Behavior**

# The Effect of Normalization on Precipitate Structure

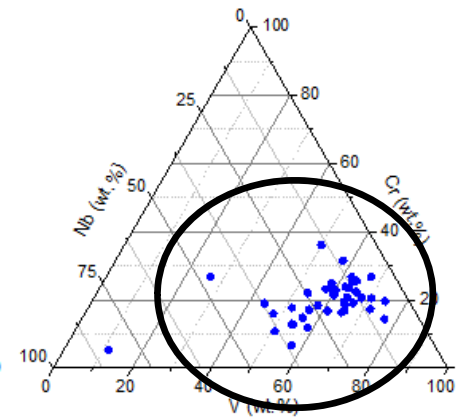
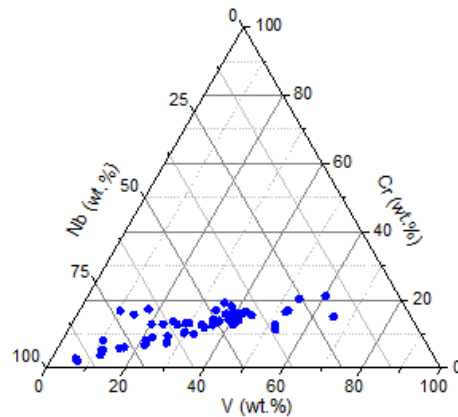
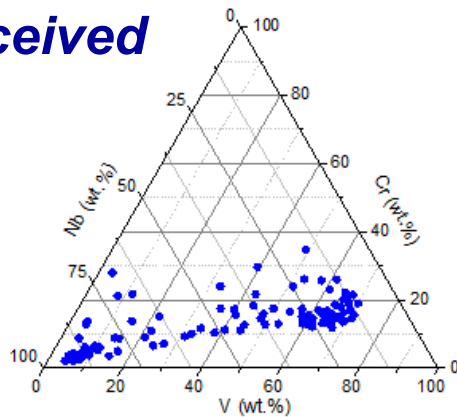
## Comparison of As-received Materials – through Optimized Heat Treatment we can make BM A more like BM C

BMA [Mean -20%]

BM B [Mean]

BM C [Mean +20%]

*As-received*



**1200°C  
Normalization**

