



ELECTRIC POWER
RESEARCH INSTITUTE

Powder Metallurgy Methods for Producing Large Nuclear and Fossil Components

*Improved & Manufacturing
Quality & Inspectability*

Presented by:

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U.S. Nuclear Regulatory Commission

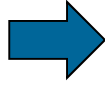
June 25, 2012

Washington, D.C.

Powder Metallurgy Methods for Large Nuclear & Fossil Components

- Today's Powder Metallurgy & Hot Isostatic Pressing?
- Why Consider Powder Metallurgy for Large Nuclear/Fossil Components?
- Technology Gaps/Barriers
- Feasibility Demonstration—Valve Body
- 316L SS Valve Body Assessment
- Grade 91 Valve Body Assessment
- EPRI Research for PM/HIP—Overview
- Key Processing Steps
- Summary

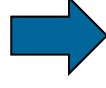
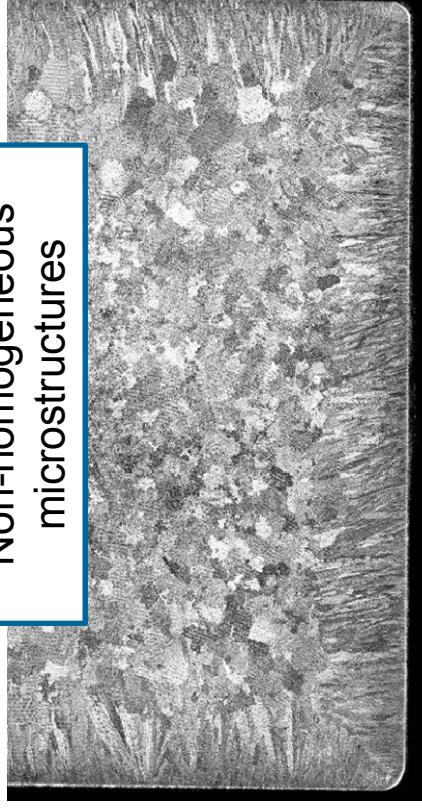
Why Powder Metallurgy??



Today's Casting
Technology

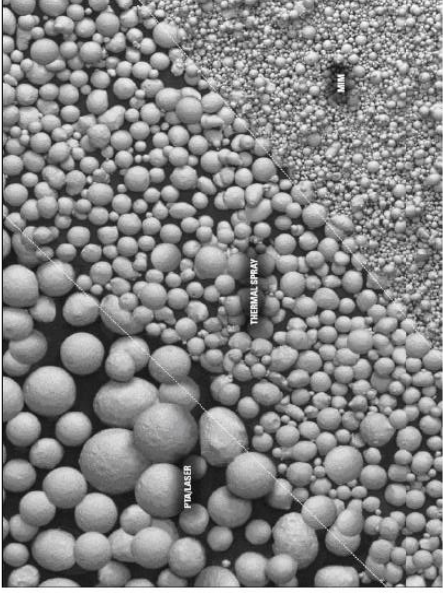


Non-homogeneous
microstructures



Poor Inspectability

Today's Powder Metallurgy & Hot Isostatic Processing (HIP)



1. Gas Atomized Metal Powders



2. Mold of Component in Can

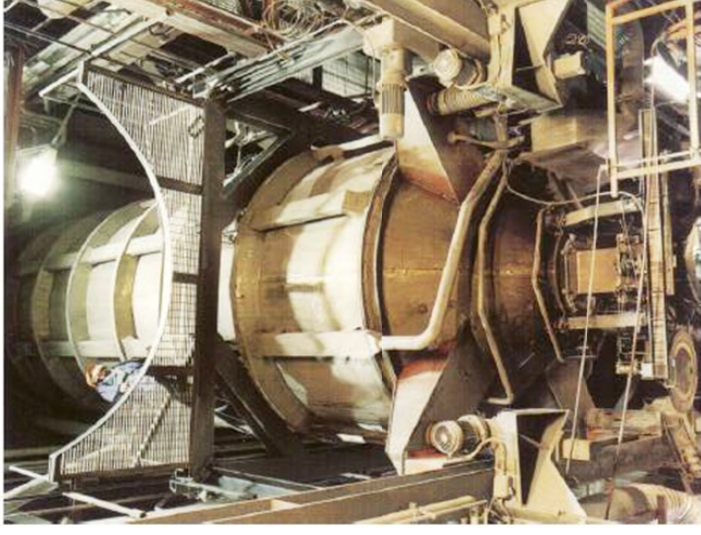
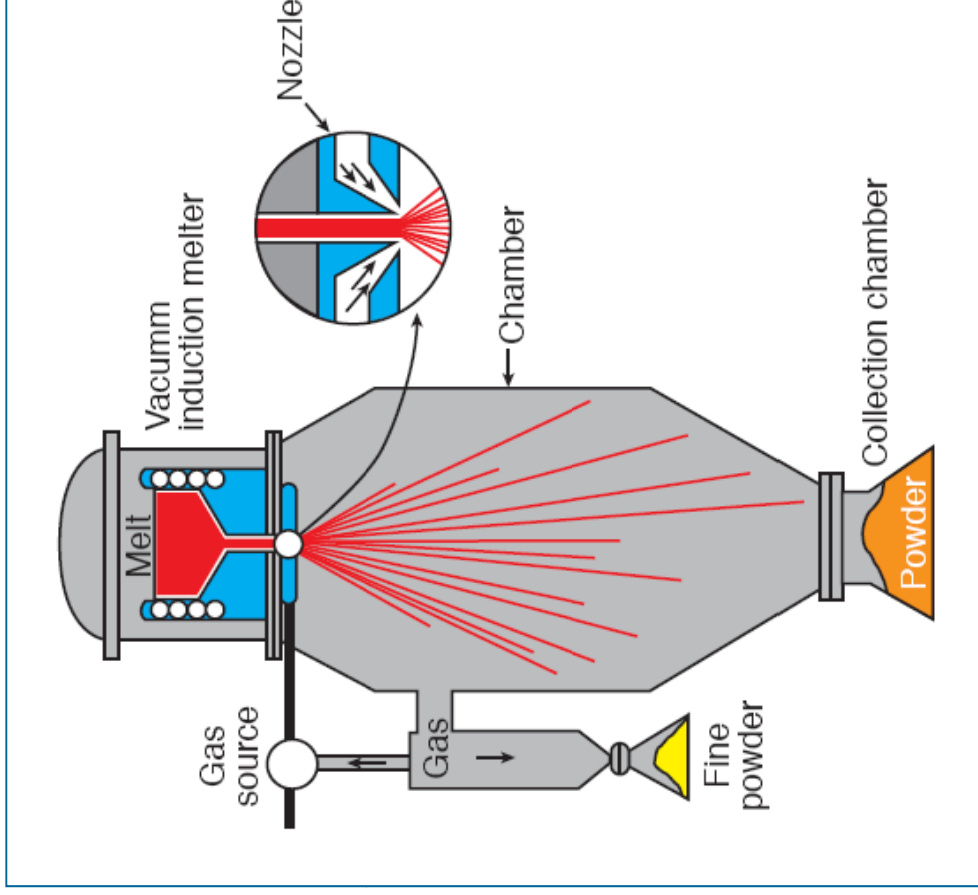


4. Final Component



3. Hot Isostatic Pressing
Apply High Pressure
($>15,000$ psi),
Temperature
($>2000^{\circ}\text{F}$)

The Key Difference.....

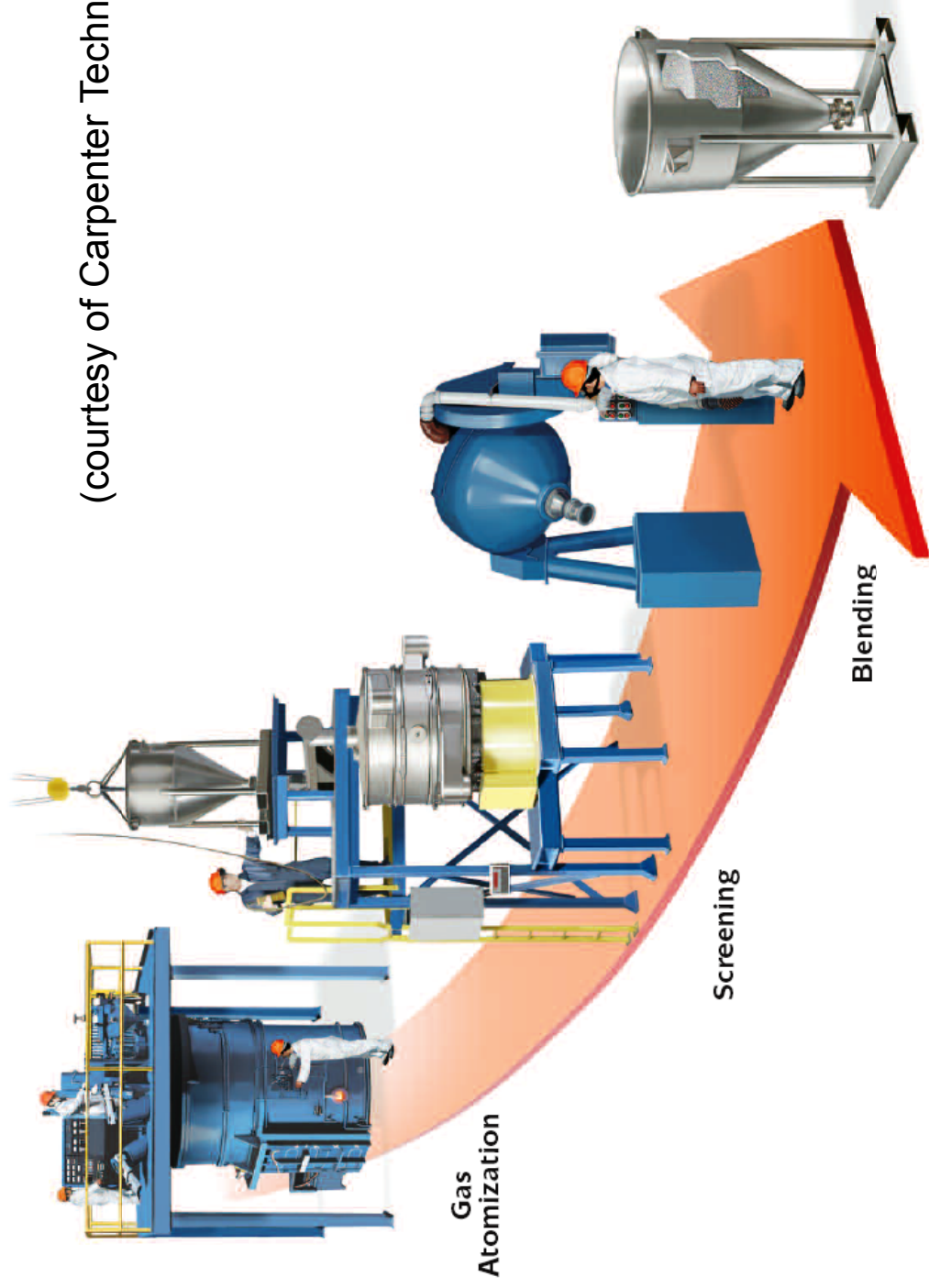


(courtesy of Carpenter Technology)

Why is Gas Atomization Important?

- No milled powders used today
- Eliminates oxides in powders & porosity in final product
- Improved packing density

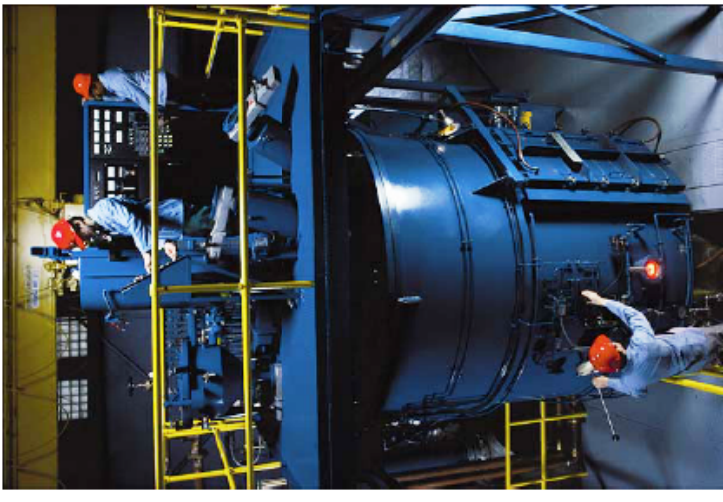
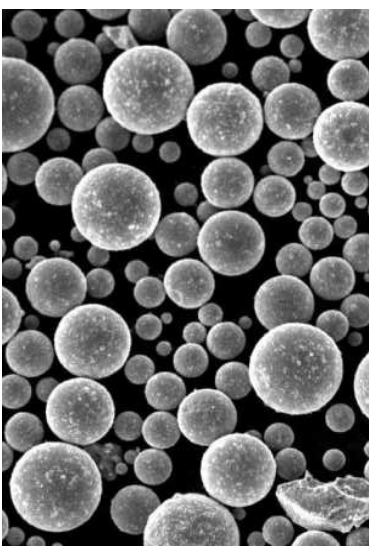
Gas Atomized Metal Powders....



(courtesy of Carpenter Technology)

Powder Consolidation (1)

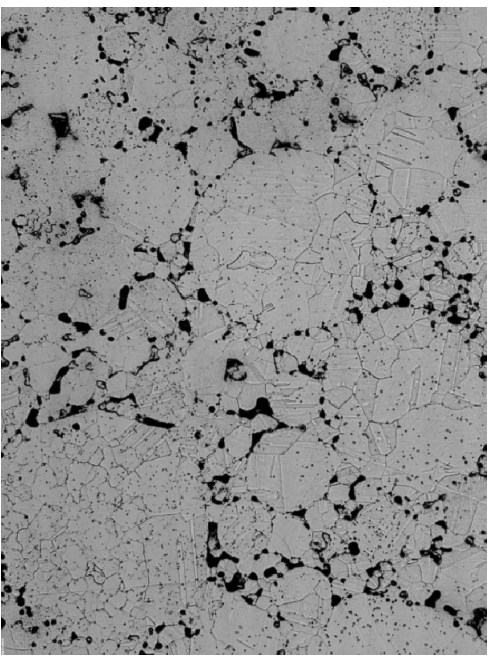
- Spherical-shaped gas atomized powders are packed into mold/container
 - A range of powder sizes are preferred.
 - smaller particles allowed to pack tightly between large particles.
- Mechanical vibration is used to pack the powder to around 70% density.
- Container is evacuated via a vacuum pump and sealed.
- After leak check, the container is placed in a HIP furnace at high pressure & temperature
 - ~2000F, ~15000 psi for stainless & nickel-based alloys



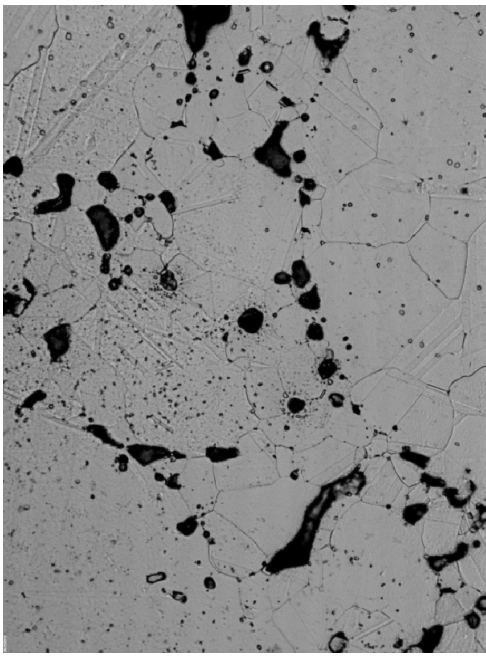
(courtesy of Carpenter Technology)

Powder Consolidation (2)

- Mechanical compaction of the powder occurs under isostatic pressure
 - Some plastic deformation of the powder particles occurs
 - reduces the void space between spherical particles
- Solid state diffusion between the particles results in necking
 - Bonding readily occurs at temperature
 - Contact is pure metal-to-metal
 - High diffusion rates
- Creep deformation is also occurring, bringing more particle surfaces into contact.



Alloy 625 – Partial Consolidation (200X)



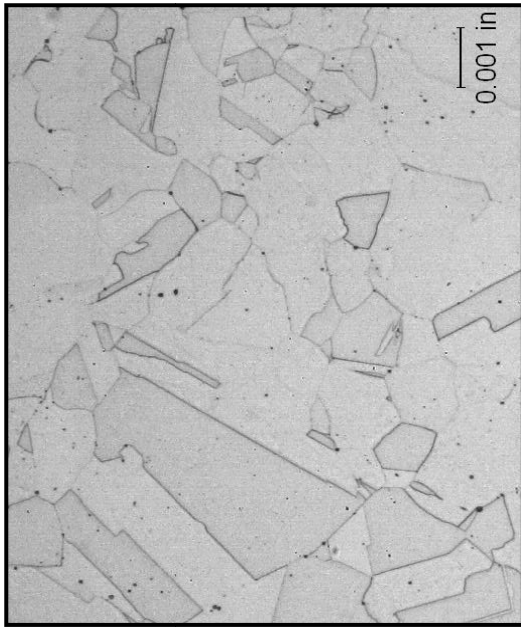
Alloy 625 – Partial Consolidation (500X)

Note: Full consolidation is achieved only with adequate HIP time and pressure!!

Powder Consolidation (3)

- As particles grow together in the final densification stage, randomly distributed voids are eliminated.
- A polycrystalline structure develops next and grain growth begins to occur.
 - Old interface boundaries are eliminated
- Final heat treatment
- Specification requires at least 99% density of the alloy in a wrought form!!!!

No powder oxides—
Eliminates earlier issues with
voiding/porosity

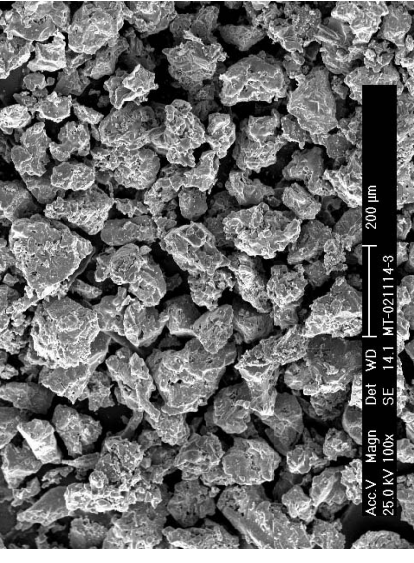


Full Consolidation
resulting in polycrystalline
microstructure – 316L SS
via PM-HIP

How Does This Differ From Earlier Powder Metallurgy Methods?

Conventional Press & Sinter Processes

- Powders are mixed with various additives and lubricants
- Cold/warm compaction, followed by sintering, results in very little densification. Part size and shape are limited.



Metal Injection Molding

- Combines metallic materials with plastic injection molding technologies
- Up to 40% binders by volume
- Sintering, dimensional tolerances are poor.

Why Consider Powder Metallurgy-HIP To Produce Pressure Components?

- ★ • Eliminates Inspectability Issues
- Enables manufacture of large, complex “*Near-Net Shape*” components
- ★ • Eliminates casting quality issues
 - Predictable production schedules
 - Significantly reduces repairs
- Enables “controlled” chemistries
- Alternate supply route for long-lead time components



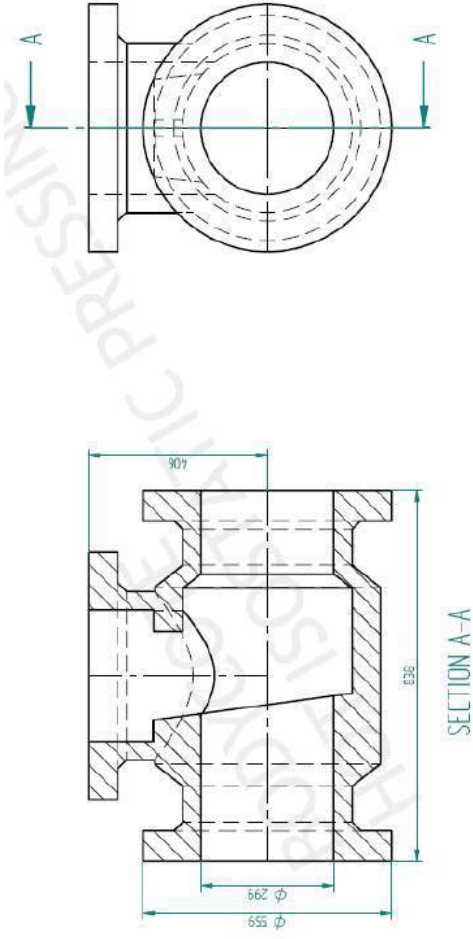
P/M-HIP Valve

Technology Gaps/Barriers



- Sizes/shapes for “*near-net shaped*” components *only recently* reached a point for consideration
 - Not currently tailored for the compositions/alloys currently of most interest to the industry.
- ASME Boiler and Pressure Vessel Code currently does not allow the use of PM produced components for the desired applications.
- Fabrication methods for some USC & oxy-combustion components presently do not exist
 - Barrier to higher temperatures/improved efficiencies

Feasibility Demonstration --12-inch Valve Body Fabrication



- Type 316L Stainless Steel Valve Body
- Near-Net Shaped
- 1716 lbs
- Annealed condition

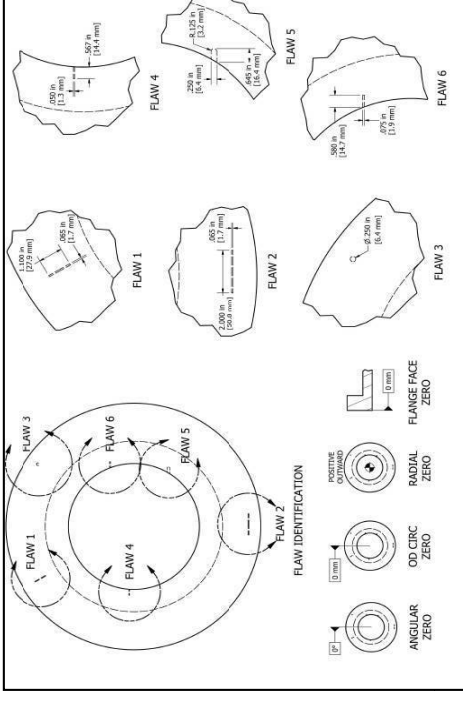
316L SS Valve Body Assessment --Inspectability



Manual and Automated UT
Scans Performed



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EDM Flaws Installed @ 6 locations
around Flange

Results

- Reflectors adequately detected and sized even thru thick (>5-inches) portions of flange
- Compared to cast 304L SS calibration blocks, the 316L PM/HIP material exhibited superior inspectability.
- No significant challenges for the UT inspection process

316L SS Valve Body Assessment

--Homogeneous Microstructure

ASTM Grain Size = 6-7

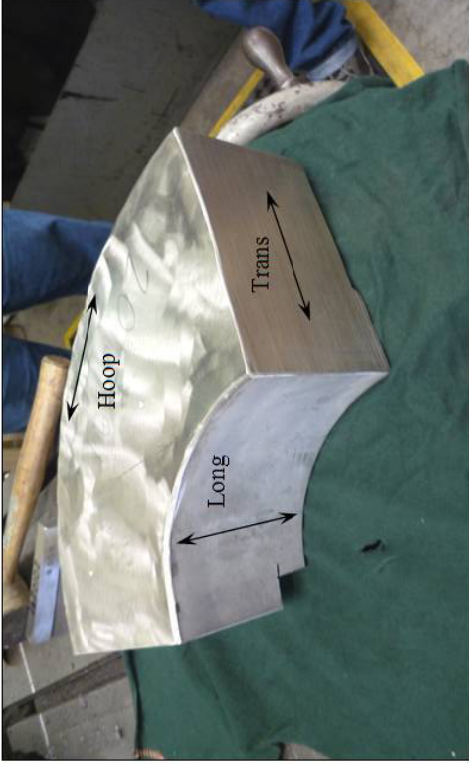
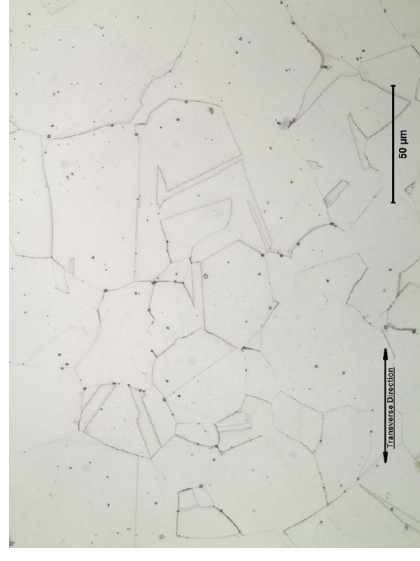
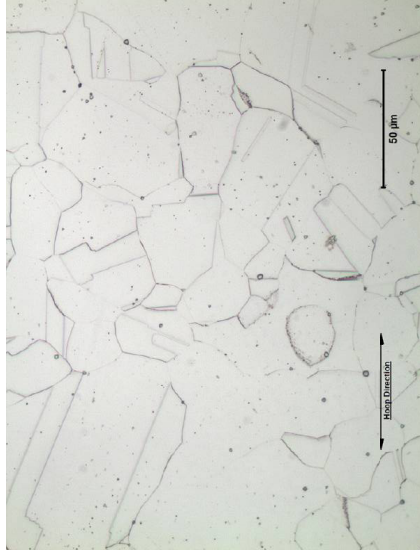
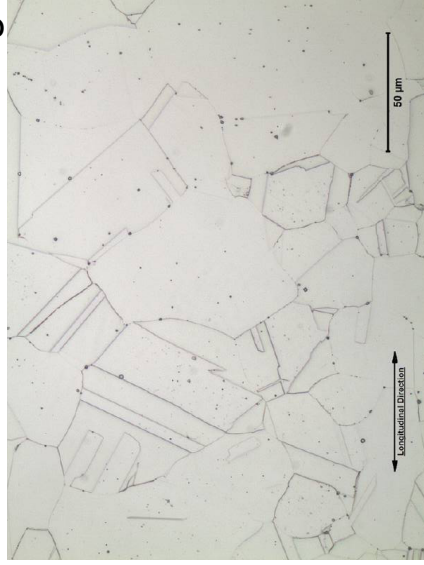


Table 1
Chemical Composition Analysis Results - (wt. %)

Element	Valve Body	316L Specification
Carbon	0.019	0.030 max
Manganese	0.99	2.00 max
Phosphorus	0.006	0.045 max
Sulfur	0.005	0.030 max
Silicon	0.73	0.75 max
Nickel	12.10	10.00 – 14.00
Chromium	17.25	16.00 – 18.00
Molybdenum	2.52	2.00 – 3.00

Section from Flange



500X Photos from 3 different orientations. Isolated carbide particles were present. No porosity

316L SS Valve Body Assessment

--Mechanical Properties (Room Temp.)

Table 2 Room Temperature Tensile Test Results				
Sample	0.2% Offset Yield Strength (psi)	Ultimate Tensile Strength (psi)	Elongation (%)	Reduction in Area (%)
A – Long	46,900	90,900	52.3	74.6
B – Long	45,300	90,400	51.4	75.3
C – Long	44,700	90,200	52.3	74.4
A - Hoop	49,800	90,800	50.6	74.6
B - Hoop	47,100	89,700	53.1	73.9
C – Hoop	49,000	90,600	50.4	74.0

Charpy Impact = >195 ft-lbs (hammer stopper)

316L SS Valve Body Feasibility

- Results very encouraging:
 - Good inspectability
 - Good tensile, yield, elongation, hardness, properties
 - Homogenous microstructure, grain size is right
 - No porosity
 - Terrific toughness
- Decided to move on to manufacture valve bodies
 - Goal: Develop ASME Code Case & Data Package
 - Assess machinability & weldability



Components Manufactured for Code Case(s)

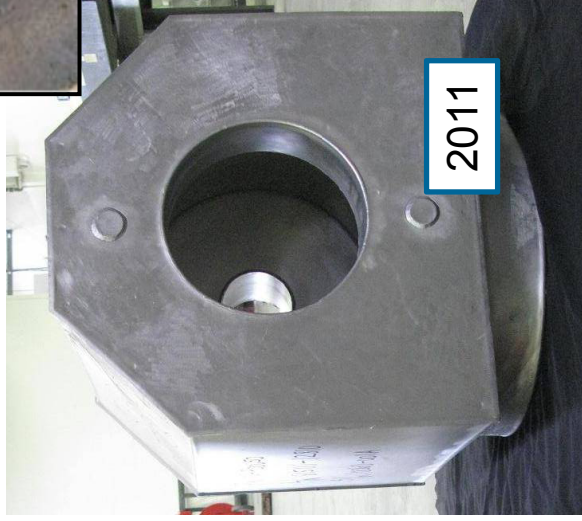
--per ASTM A988-11



3- Grade 91 & 1- IN625
Valve Bodies (Carpenter)



316L SS Tee (Rolls-Royce)



3 – 316L SS Valve Bodies
(Carpenter)

ASME Code Case Development

- Chemical Composition
- Grain Size
- Drawings & Images
- Microstructure
- Density & Inclusion Content
- Tensile/Yield Properties to 1000F
(& stress/strain curves)
- Toughness
- Fatigue
- Weldment Properties

ASME Backlog

Chemical Composition—4 Heats

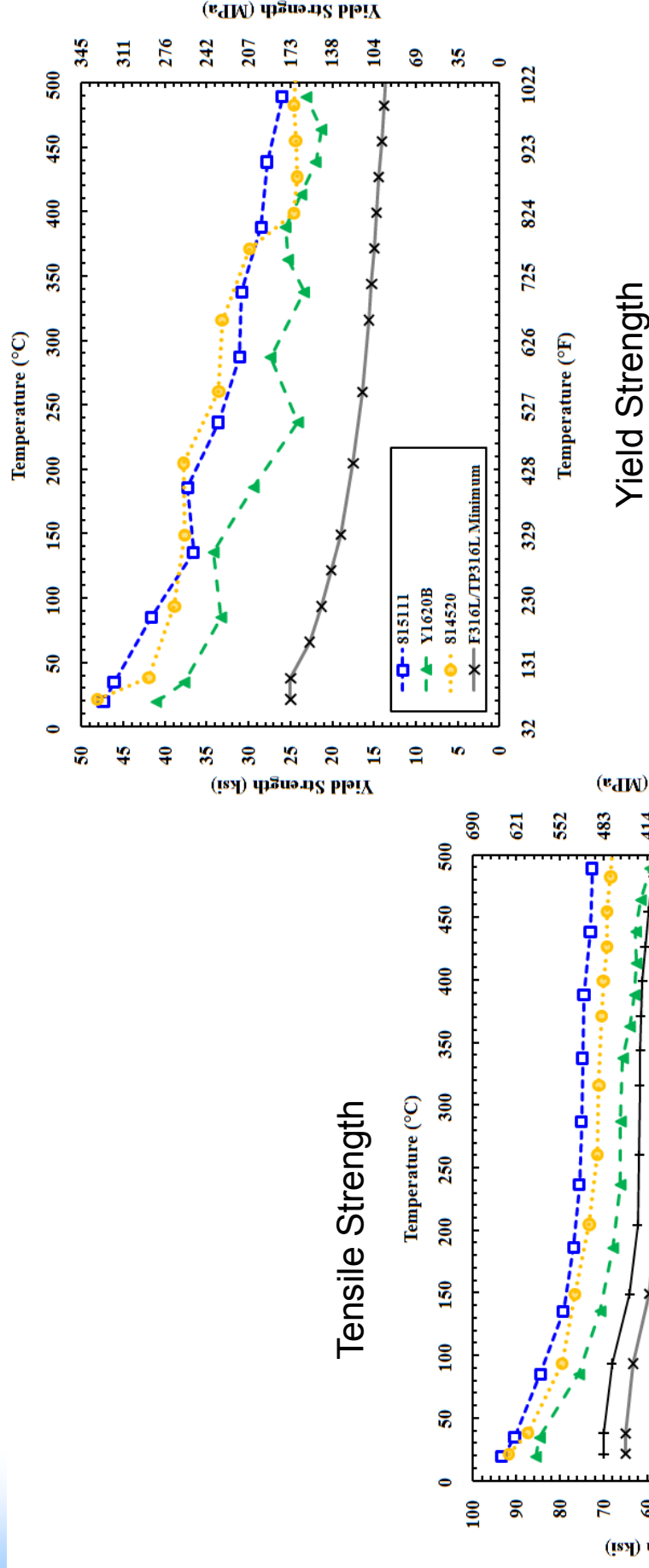
-- S31603 (316L SS)

Heat	S31603 (316L) Specification ¹		814520	815111	Y1620B	33836-01A
Size			1892 lbs	1200 lbs	1200 lbs	NS ²
Product Form			Valve	Valve	Valve	Tee Piece
C	0.030 max		0.013	0.021	0.004	0.022
Mn	2.00 max		0.90	0.95	1.41	1.76
P	0.045 max		0.014	0.009	0.005	0.016
S	0.030 max		0.003	0.004	0.005	0.006
Si	1.00 max		0.70	0.80	0.55	0.32
Ni	10.0-14.0		12.1	13.2	13.3	12.10
Cr	16.0-18.0		17.3	17.3	16.87	17.80
Mo	2.0-3.0		2.55	2.50	2.37	2.50
N	0.10 max		0.10	0.14	0.038	0.090
Others	N/A		O: 0.010	N/A	N/A	O: 0.0140 Cu: 0.020 Co: 0.040

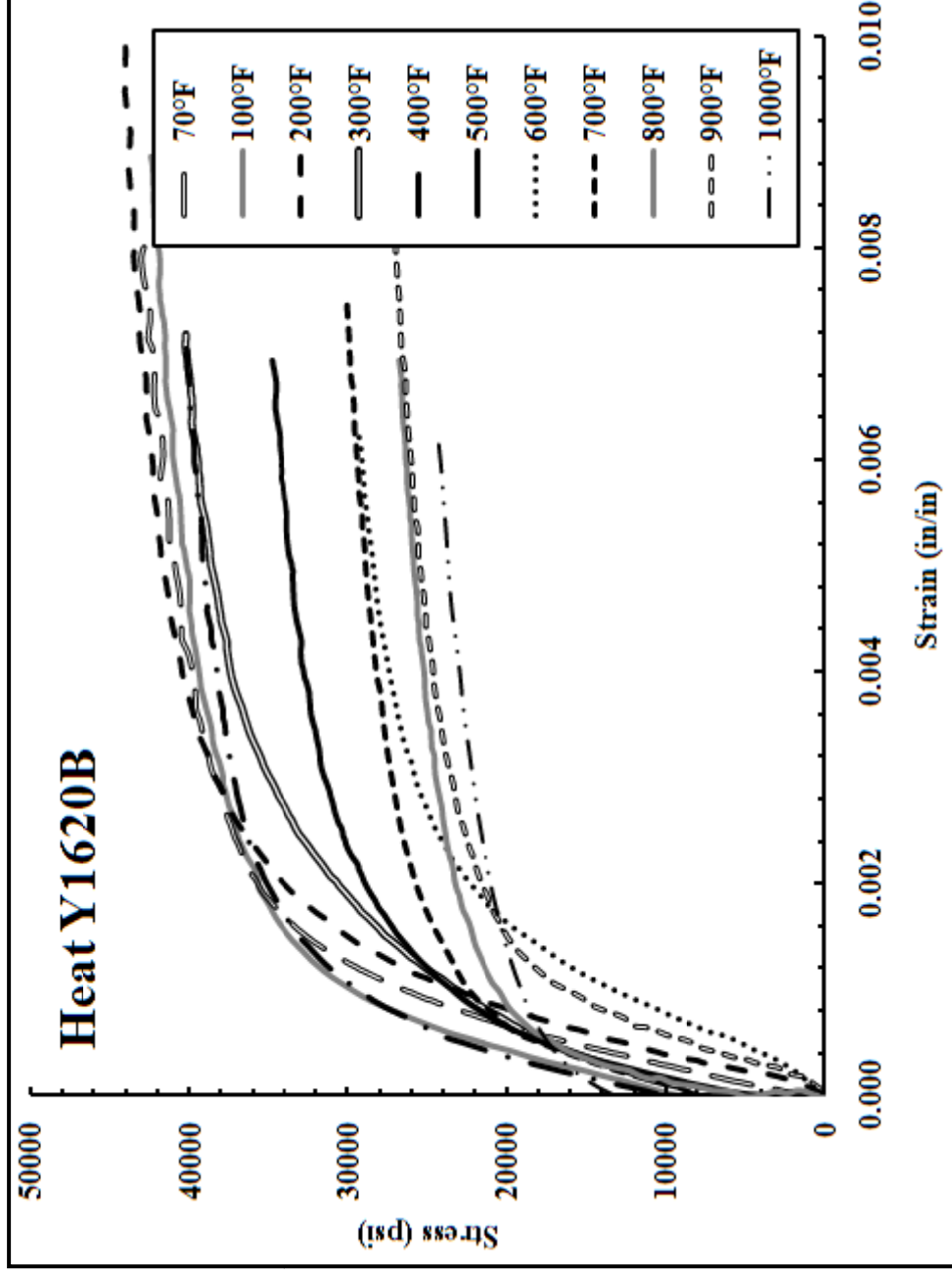
¹Specification = ASTM A988/A988M

²NS = Not Specified

Tensile & Yield Strength – 316L SS (for Code Case)

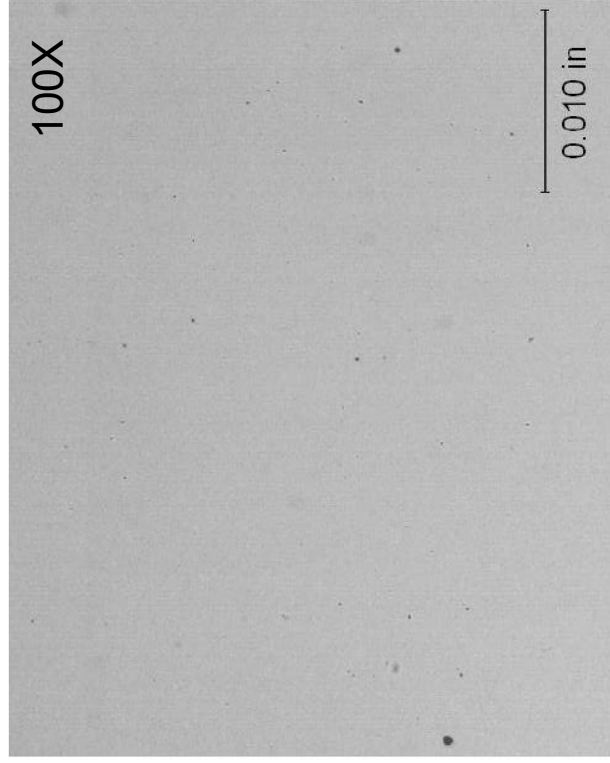


Representative Yield Stress-Strain Curve

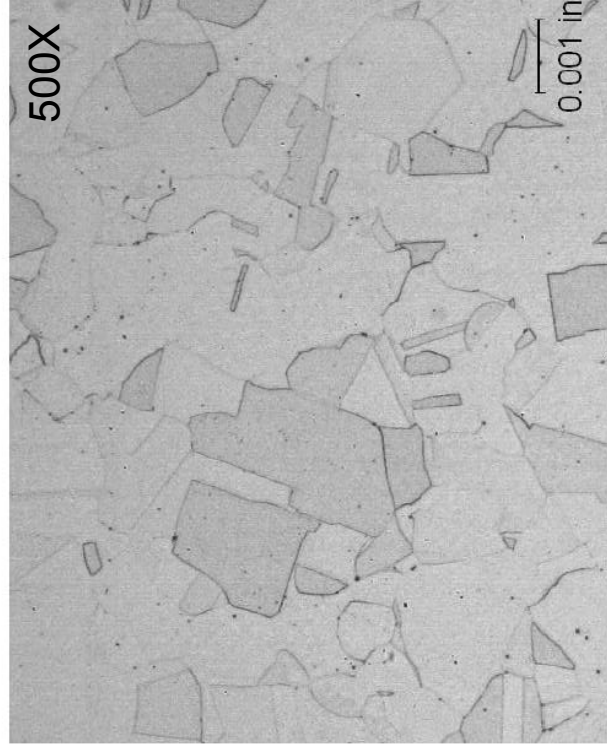


316L SS Mechanical Properties

- Charpy Impact: >122 ft-lbs (3 orientations)
- Grain size: ASTM 5.5 to 7.0
- ROA ~ >68%; Elongation > 40% (Room temp)
- Inclusion content (per ASTM E45): zero, (3 orientations)
- Density measurements: No porosity observed (3 orientations)

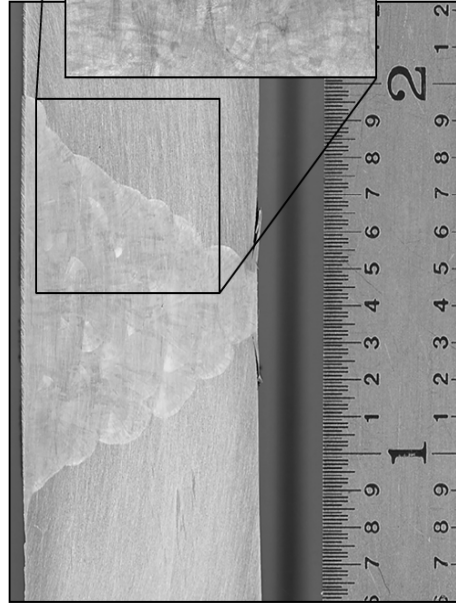


As-Polished

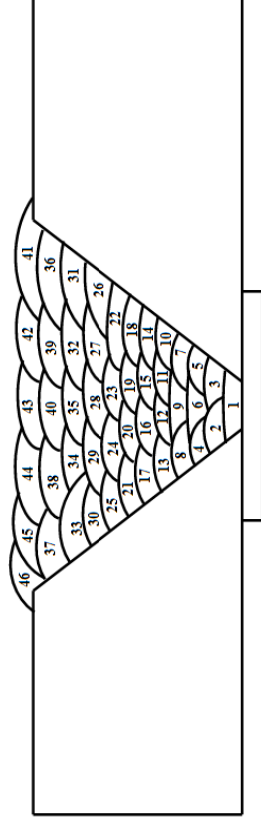
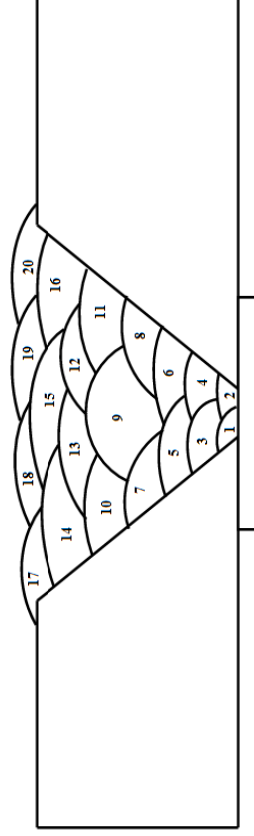


Etched Kalling's Reagent

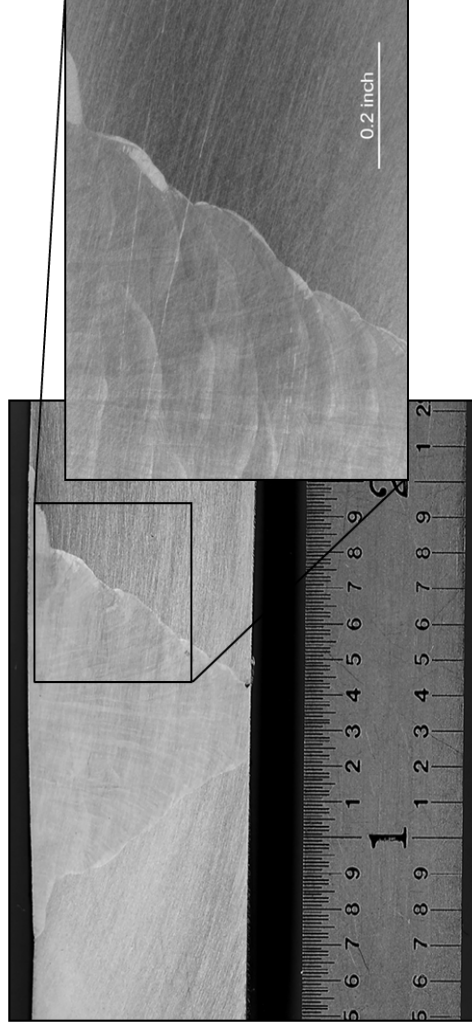
316L Weldments for Code Case



GTAW



0.75" plate; 30° bevel



SMAW

316L Weldments for Code Case

Room Temperature Tensile Test Results

Process	Specimen	TS (ksi)	TS (MPa)	YS (ksi)	YS (MPa)	Elong. in 2" (%)	Fracture Location
SMAW	2	94.7	653.1	60.8	419.3	27.5	Weld
	5	95.1	655.9	60.4	416.6	28.0	Weld
	7	94.1	649.0	58.4	402.8	28.0	Weld
GTAW	2	83.9	578.6	51.2	353.1	36.0	Base
	5	84.2	580.7	50.7	349.7	38.5	Base
	7	84.8	584.8	52.9	364.8	39.0	Base

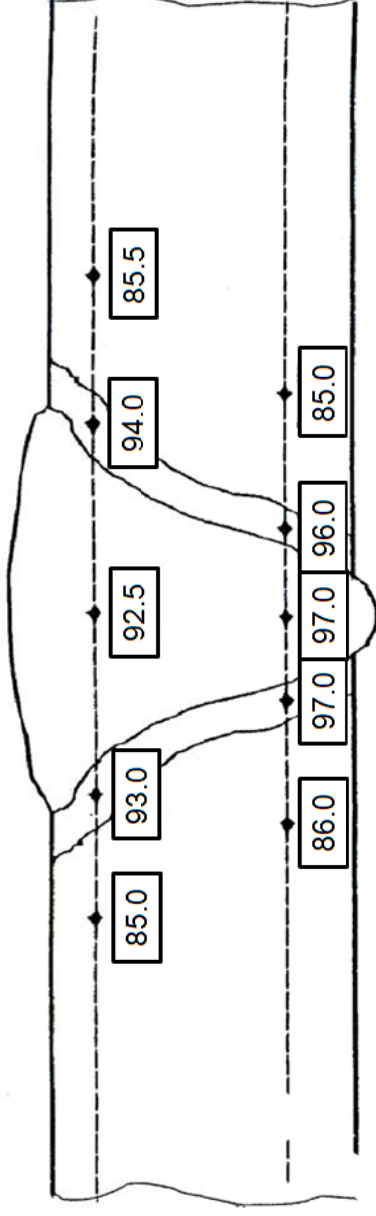


2T Side Bend Tests, passed--SMAW

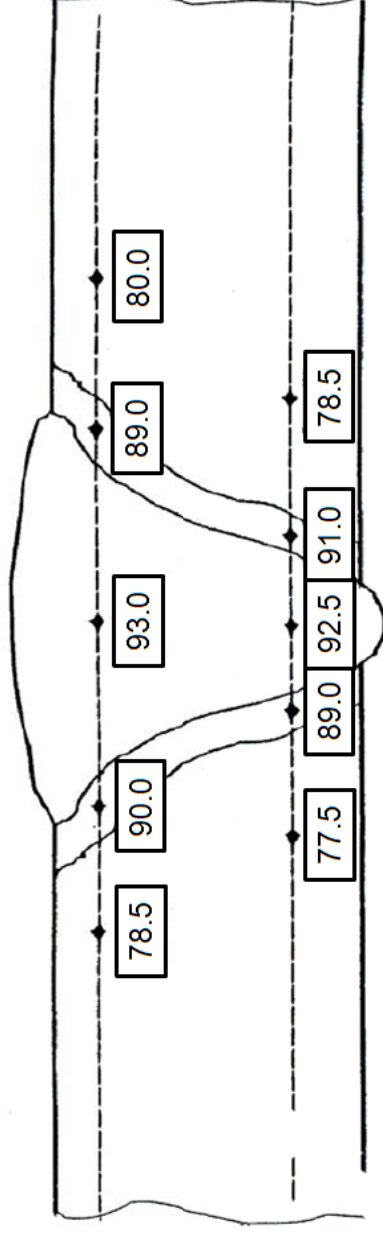


2T Side Bend Tests, passed--GTAW

316L Weldments for Code Case

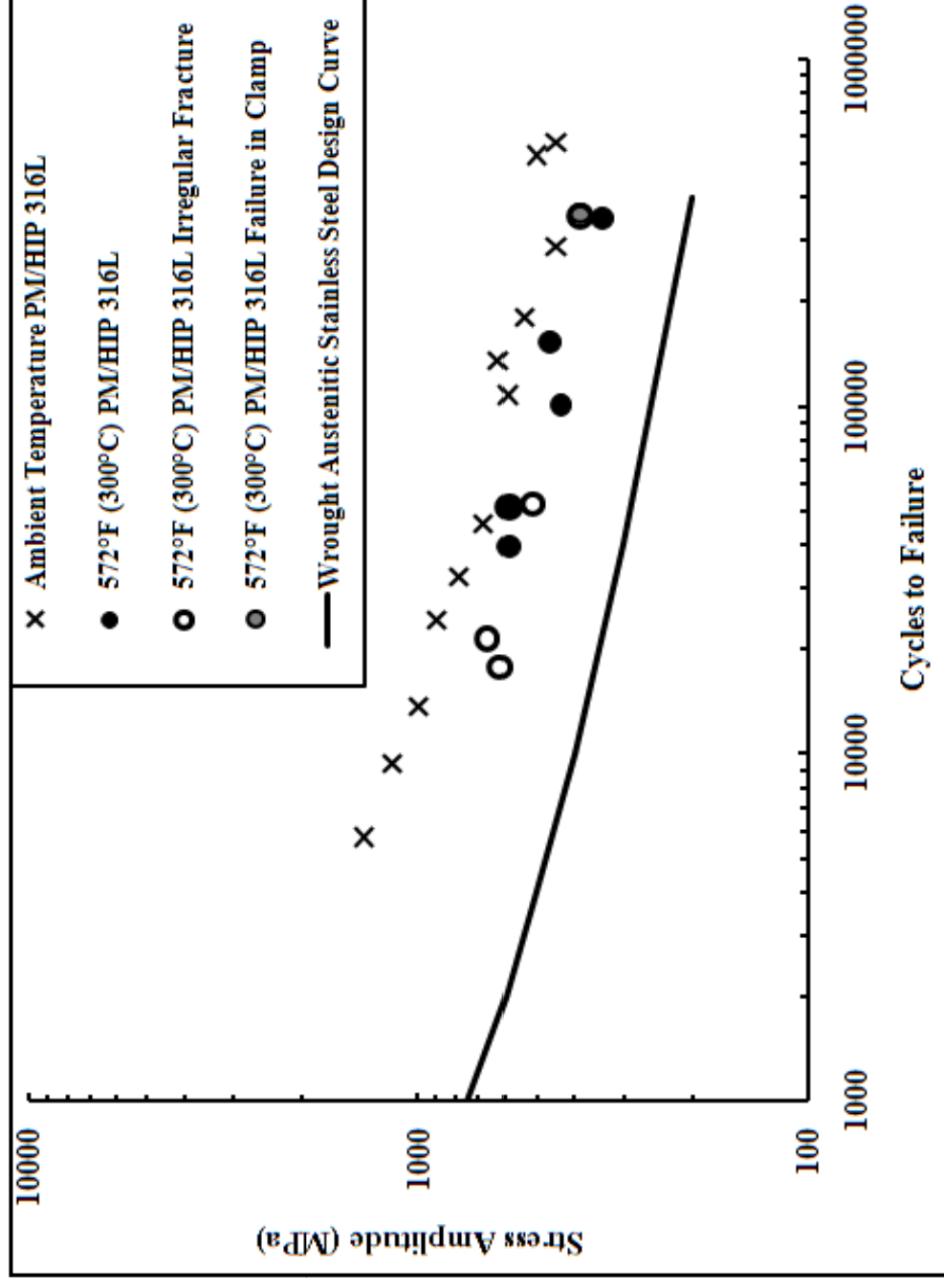


SMAW Weldment, Hardness Profile (Rockwell B)

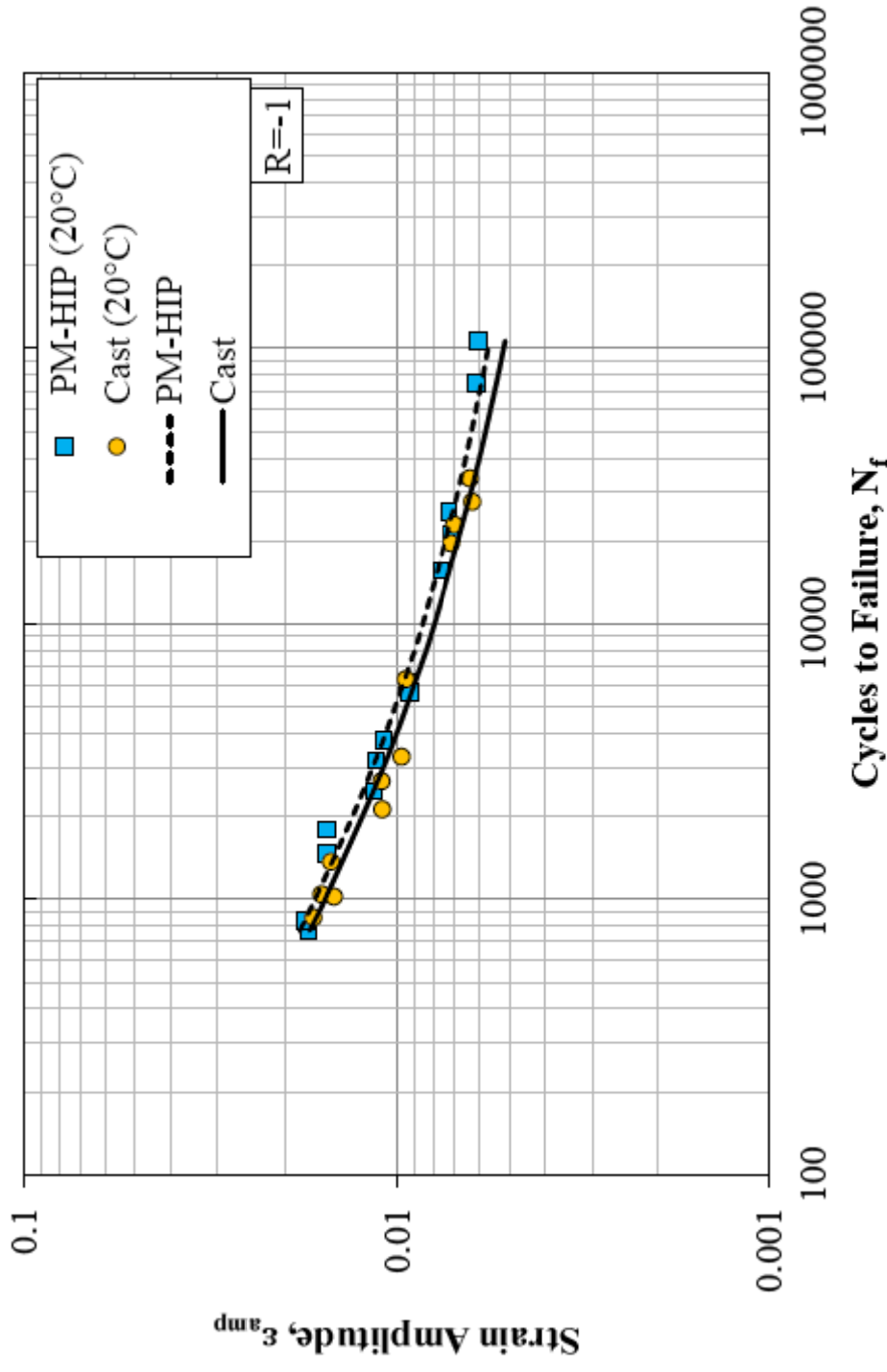


GTAW Weldment, Hardness Profile (Rockwell B)

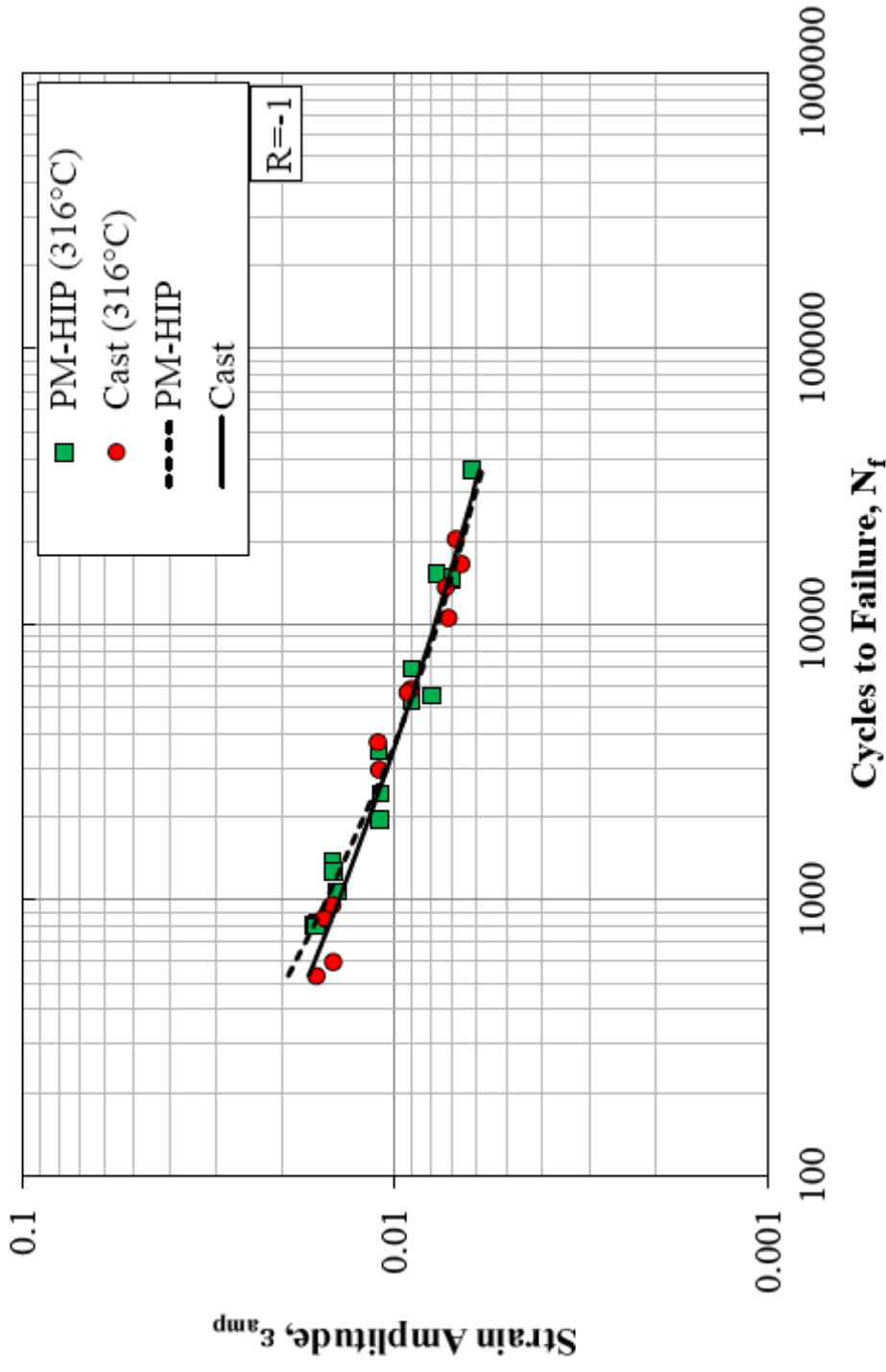
316L SS PM/HIP Fatigue Data (courtesy of Rolls-Royce)



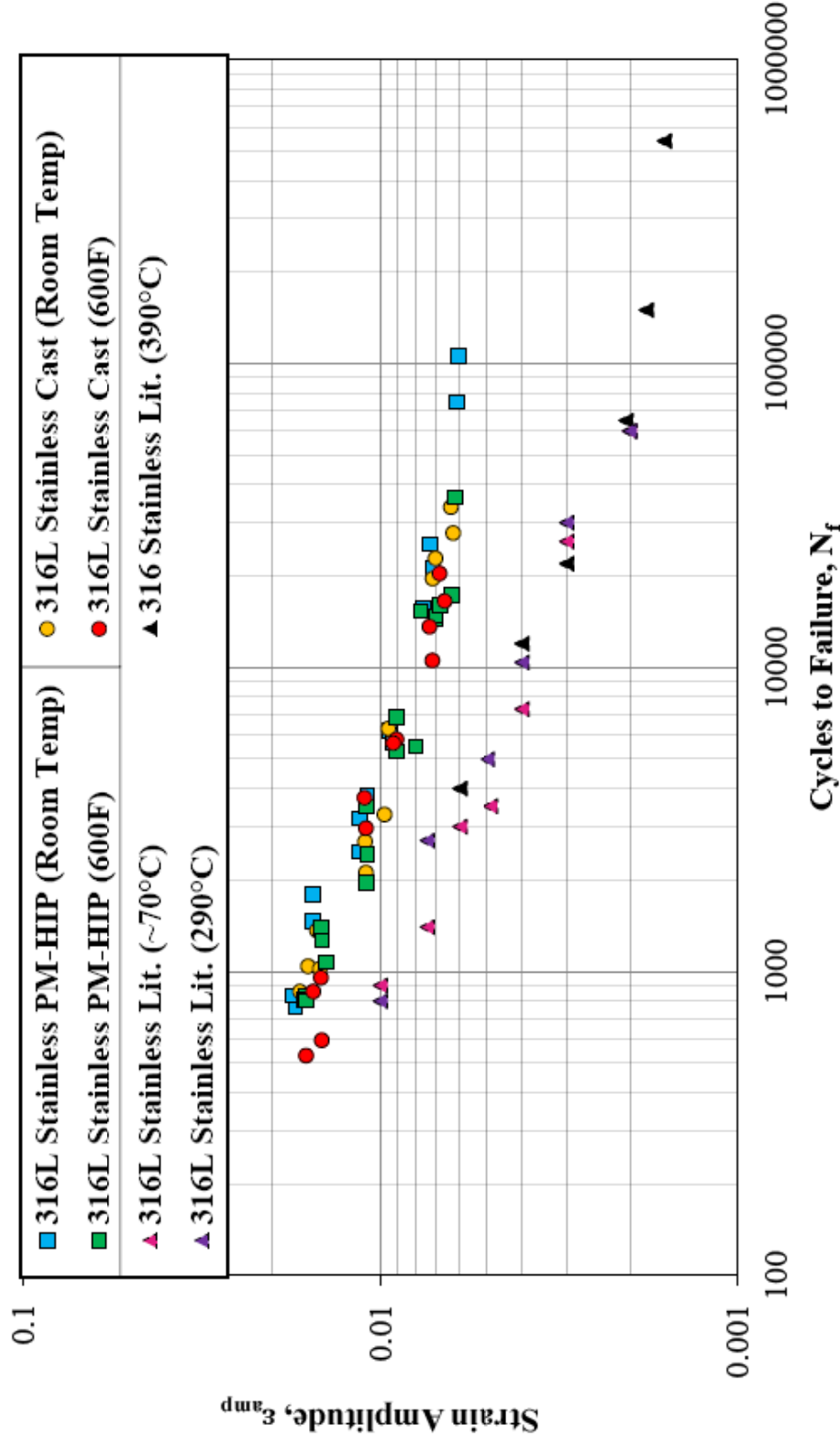
Fatigue Data: PM-HIP vs. Centrifugal Cast --316L SS (Room Temp)



Fatigue Data: PM-HIP vs. Centrifugal Cast --316L SS (600F/316C)

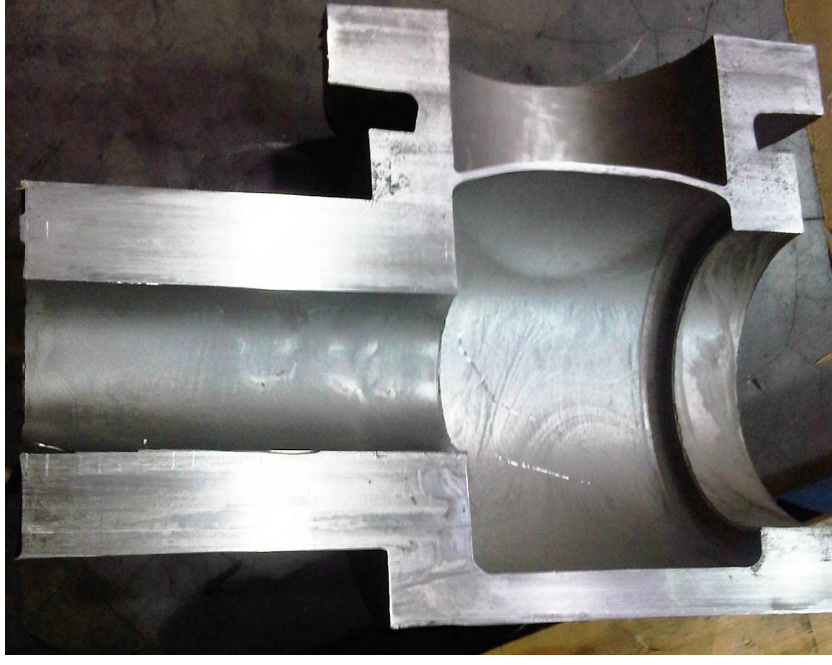


Fatigue Data: PM-HIP & Centrifugal Cast --316L SS (compared to NRC data)



Chopra, O. K. "Mechanism and Estimation of Fatigue Crack Initiation in Austenitic Stainless Steels in LWR Environments." U.S. Nuclear Regulatory Commission (2002): 6.

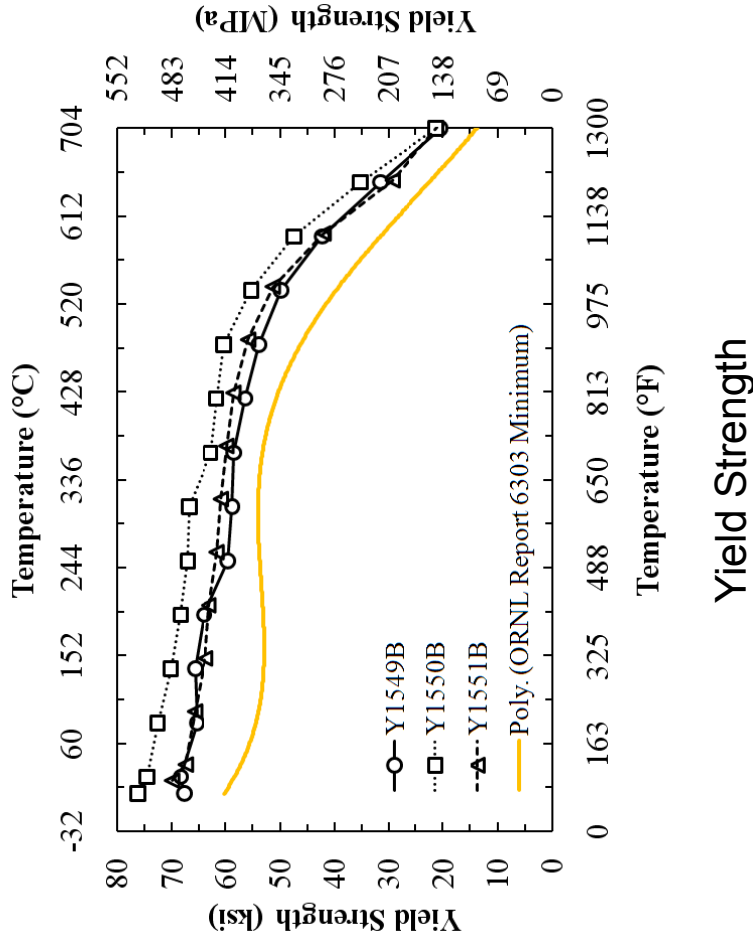
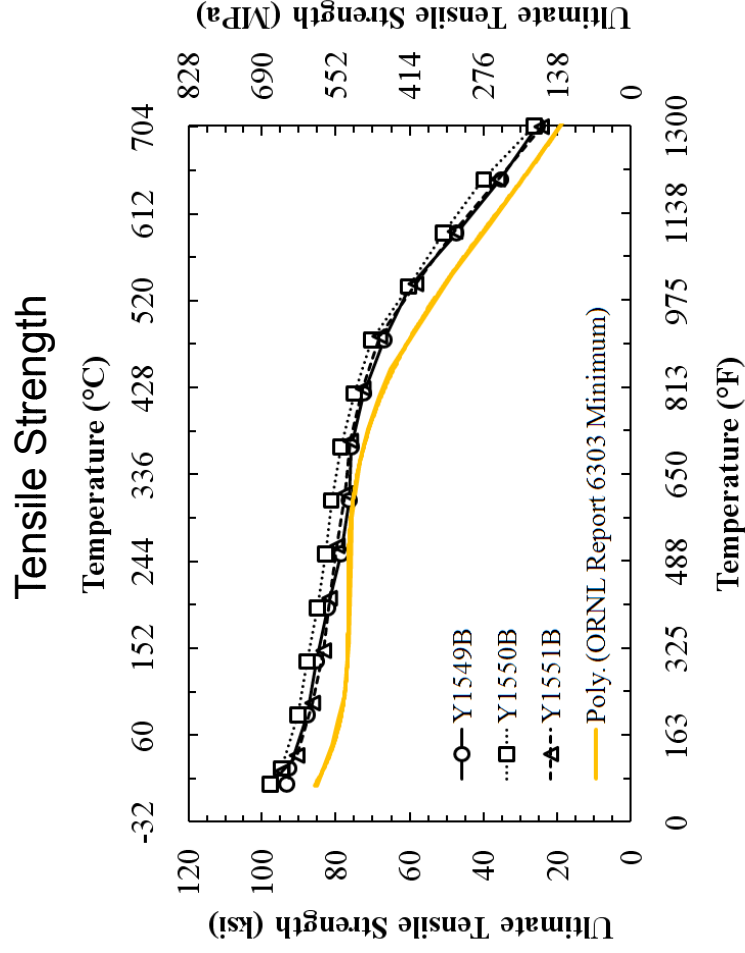
Grade 91—3 Valve Bodies Manufactured



Grade 91 Chemical Composition—3 Heats

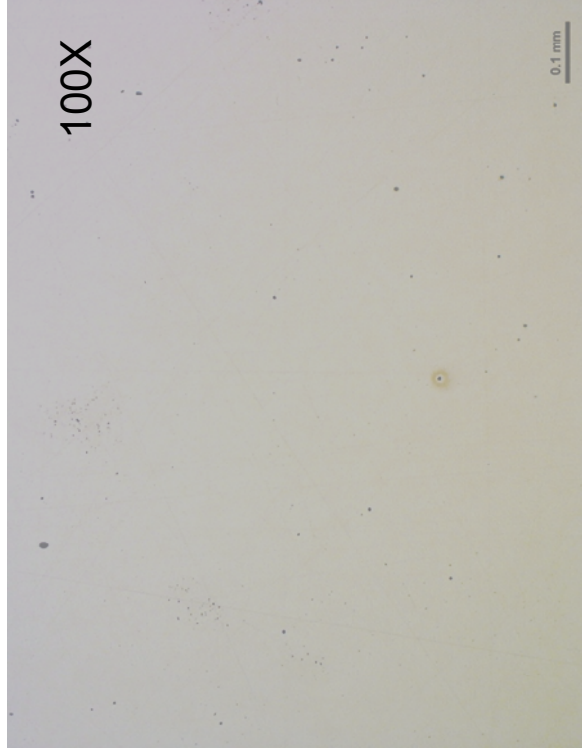
Heat	Grade 91 Specification		Y1549B ^{2,3}		Y1550B ^{2,3}		Y1551B ^{2,3}	
	UNS K91560 ¹	EPRI	Cert	Ind. Analysis	Cert	Ind. Analysis	Cert	Ind. Analysis
C ^A	0.08-0.12		0.09	0.11	0.08	0.10	0.09	0.11
Mn	0.30-0.60		0.44	0.46	0.44	0.46	0.42	0.45
P	0.020 max.		0.002	0.002	0.006	0.01	0.003	0.008
S	0.010 max.		0.009	0.007	0.008	0.007	0.008	0.007
Si	0.20-0.50		0.20	0.30	0.24	0.36	0.22	0.33
Cr ^B	8.0-9.5		8.88	8.99	8.42	8.81	8.12	8.46
Mo	0.85-1.05		0.93	0.92	0.88	0.88	0.96	0.90
V	0.18-0.25		0.25	0.25	0.25	0.25	0.22	0.23
Cb	0.06-0.10		0.08	0.08	0.08	0.08	0.07	0.08
Ni	0.40 max	0.20 max	0.08	0.08	0.08	0.08	0.09	0.08
N ^A	0.030-0.070	0.035-0.070	0.04	0.0428	0.04	0.0397	0.04	0.0416
Al	0.040 max.	0.020 max.	<0.01	<0.002	<0.01	<0.002	<0.01	<0.002
Ti		0.010 max.	<0.01	0.002	<0.01	<0.002	<0.01	<0.002
Zr		0.010 max.	<0.01	<0.002	<0.01	<0.002	<0.01	<0.002
Cu ^C		0.25 max.		0.03		0.03		0.03
As ^C		0.012 max.		<0.002		<0.002		<0.002
Sn ^C		0.010 max.		0.002		0.002		0.002
Sb ^C		0.003 max.		<0.001		<0.001		<0.001
As+Sn+Sb+Pb	< 0.010			<0.005		<0.005		<0.005
N/Al Ratio	4.0 min.			>21.4 ⁴		>19.85 ⁴		>20.8 ⁴

Grade 91 Tensile & Yield Strength Data

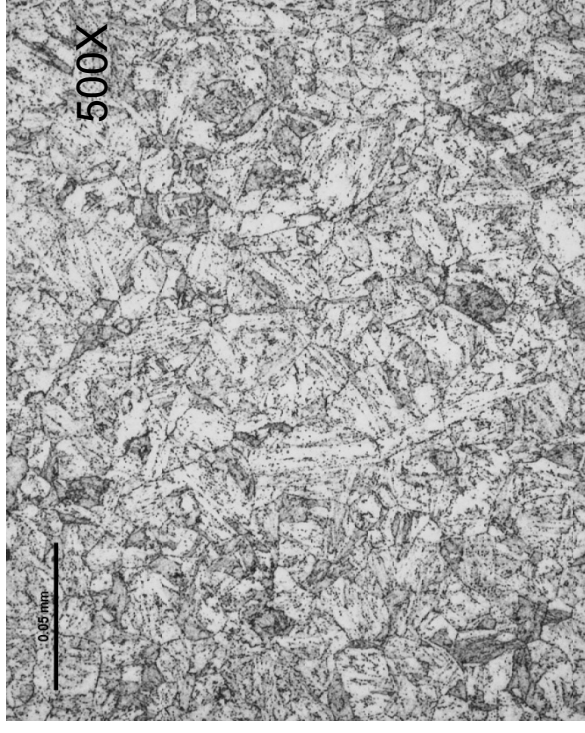


Grade 91—General Properties

- Toughness – >91 ft-lbs; Hardness – 92 to 94 Rb
- Grain size – ASTM 7.5 to 8.0
- ROA ~ >68%; Elongation >25% (Room temp)
- Inclusion content (per ASTM E45): zero, (3 orientations)
- Density measurements: a few isolated pores observed

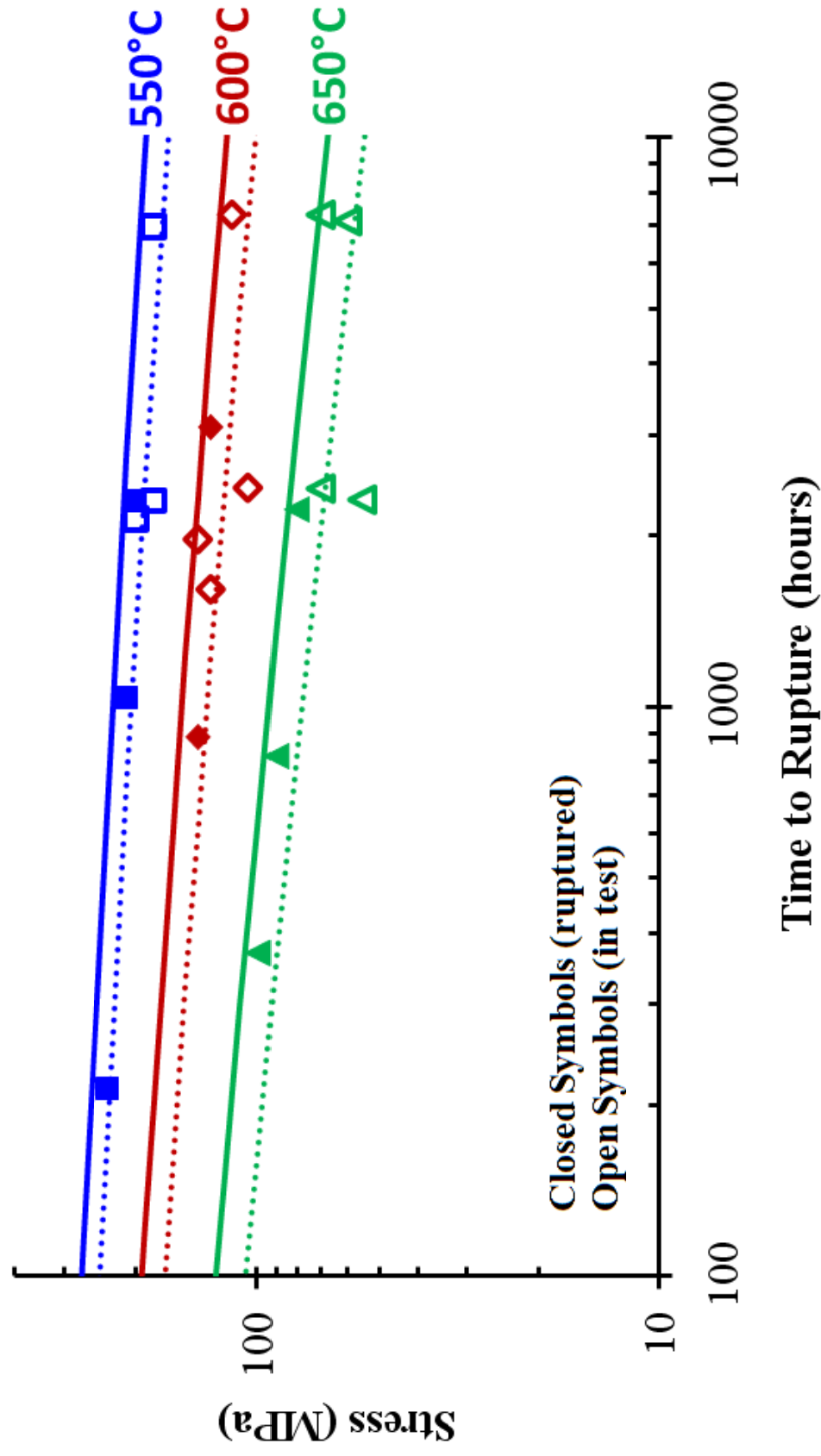


As-Polished

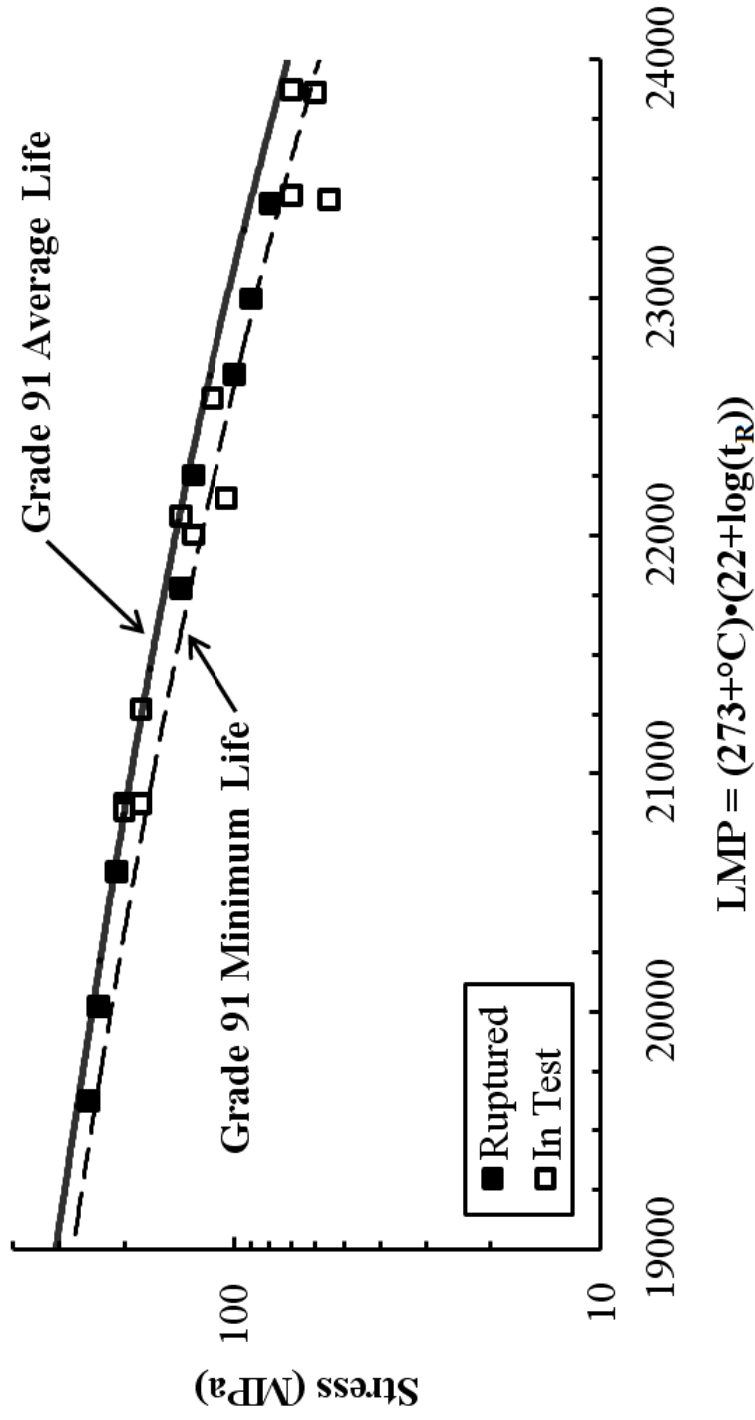


Etched

Grade 91 Creep Rupture Data



Grade 91 Creep Rupture Data



EPRI Research In PM/HIP

Development of 2 ASME Code Packages

- 316L SS (4 heats)— submitted November 2011
- Grade 91— target 4Q 2012
- Working with Carpenter, Rolls Royce, Dresser, & Tyco-Crosby Valves

Feasibility Assessments

- SA508 Class 1 – new manufacturing technology for vessel components?
- Alloy 690 tubes – improved microstructures
- IN625 & Haynes 282 – for USC and Oxy-combustion components

Erosion Protection (via Functionally-Graded Compositional Controls)

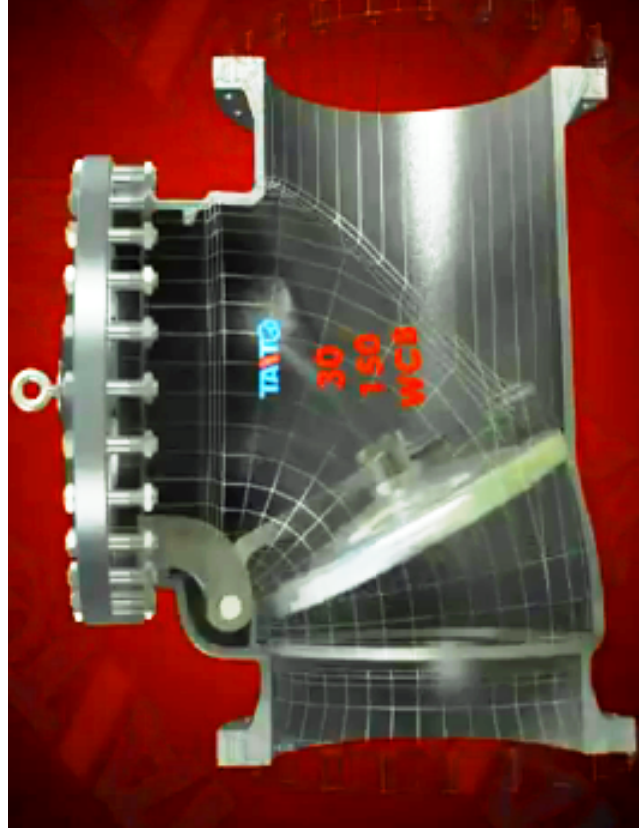
- Development of hard-facing alternatives for valves, blades, valve stems, fans, nozzles, coal mills, etc.
- Erosion/galling/wear applications for nuclear (2012), fossil (2014) & USC (2014)
- 4-year research effort; Also useful for corrosion protection

Erosion-Resistant Surfacing Alloy Development

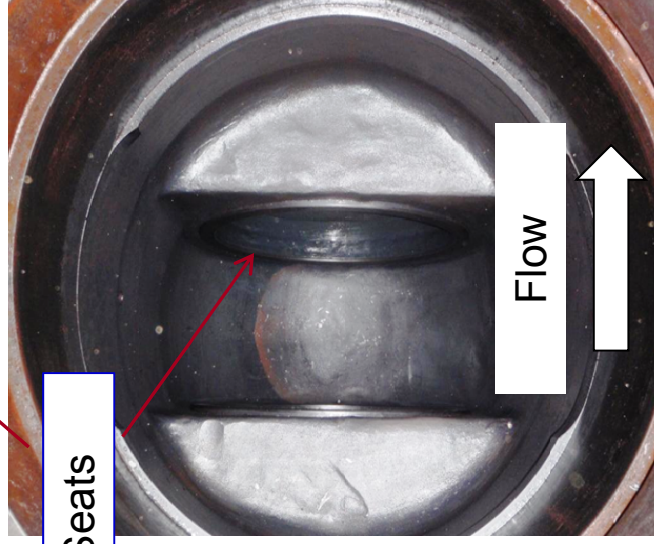
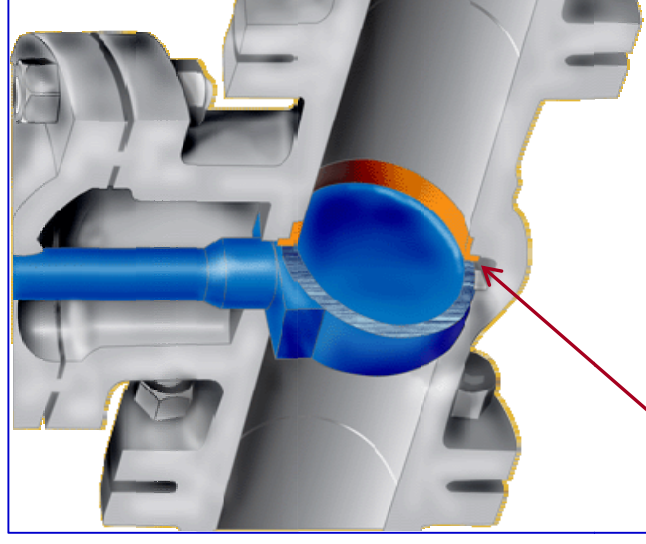
- Design “targeted” properties for specific service
- Modeling of Surfacing Compositions
- Evaluating “graded” coupons

Co-free Hard-facing
75% / 25%
50% / 50%
25% / 75%
316L Component

Functionally Graded Alloys



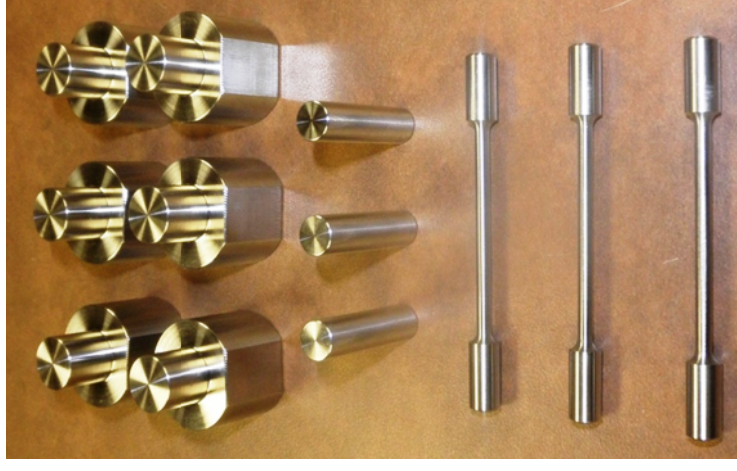
Valve Seat Damage --Example



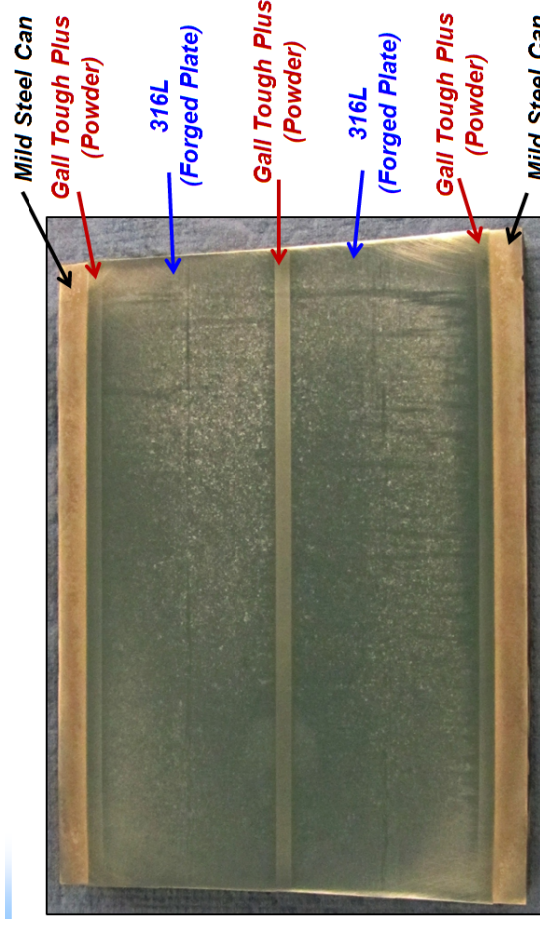
Erosion-Resistant Surfacing Alloy Development



P/M-HIP parts and
tests specimens



Alloy modeling &
development

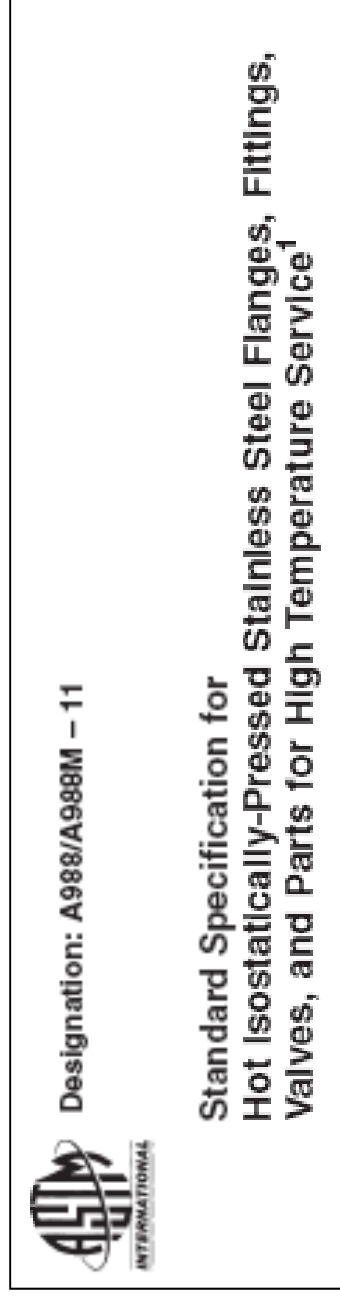




Key Processing Steps for PM-HIP

Key Processing Steps (1)

- As with any material fabrication process, there are basically two types of controls that can be applied:
 - In-process controls
 - End-product controls
- From a purchaser's viewpoint, controls applied by a specification producing an end-product are desirable:
 - eg., Chemistry, mechanical properties, density, NDE, microstructure



Key



- PM-HIP should follow same path, using in-process controls to achieve end-product requirements

Key Processing Steps (3)

--ASTM A988-11



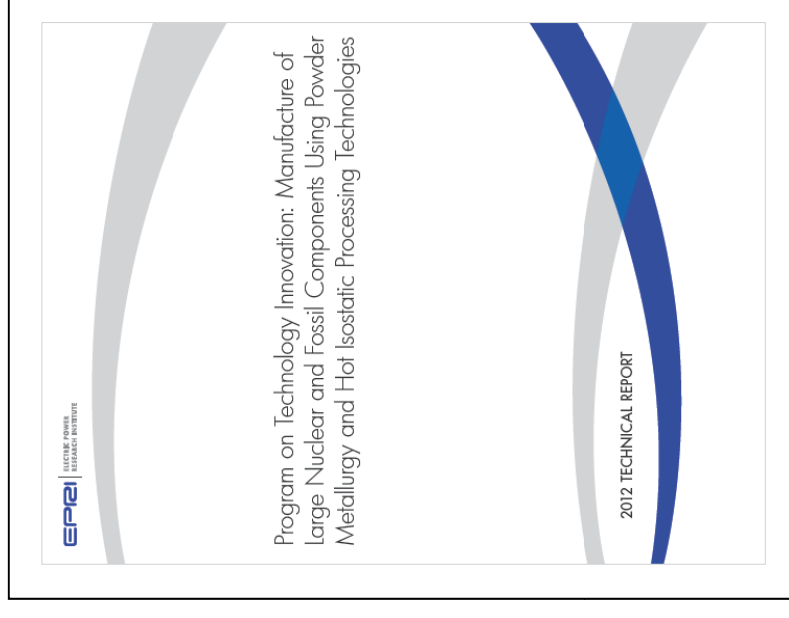
- Materials & Manufacture
- Chemical Composition
- Heat Treatment
- Structural Integrity (density, microstructural, hydrostatic)
- Mechanical Properties
- Corrosion Testing
- Product Analysis
- Repair
- Inspection

Covers several grades of stainless steels:
Martensitic, austenitic, age-hardenable,
and austenitic-ferritic stainless steels for
use in pressure systems

Key Processing Steps (4)

--Process and Product Controls

- EPRI Report 1025491 – **Appendix A** covers the process steps (reproduced in Handout):
 - Powder Production
 - Canning
 - Hot Isostatic Processing
 - Post-HIP Processing & Inspection
- ASTM A988-11 – end-product quality achieved:
 - Microstructure, mechanical & corrosion properties, inspection, composition



Summary

- Homogeneous microstructures
- ★ • Excellent inspectability
- 10-15% improvement in mechanical properties over cast components
- Once detailed drawings are available, components can readily be produced
 - rapidly and multiple times
 - Near Net Shape
- ★ • Meets production schedules
 - Significant reduction in repairs as compared to cast components (valves, flanges, pump housings, shells, etc)
- What's Next—Grade 91 (late 2012)



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