



NETL Advanced 9% Cr Steel: Update and Current Development Status

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Introduction & Background



- Reliable materials for energy systems require effort to understand the relationship between the disposition of elements, leading to stable multi-length scale structural features that resist change over long times under very severe, and ever changing, environmental conditions.
- Complementary to this is proficiency in manufacturing these materials using relevant melting, or other, techniques that attain the desired structural features for requisite mechanical / physical performance consistent with the application.
- However, to integrate these new materials into future FE energy systems depends on the continued evolution of computational materials models, integrating them into alloy design, manufacturing and life prediction with the focus on real microstructures that can be described by a physics framework for their entire life.
- And yes. We want to do all this as cheaply as possible, using, if possible, existing infrastructure and processes!



- Ferritic/Martensitic Cr steels form the backbone of current steam delivery systems.
- These alloys are less expensive to produce & in general can be recycled.
- □ CrMoV, NiCrMoV & steels with < 5% Cr make up the majority in tonnage in steam power plants operated < 570°C.
- In the hotter sections of the boiler & steam turbine, i.e., temperatures greater than 570°C, advanced 9-12% Cr steels will need to be used.
- At the current time, <u>620°C</u> is the approximate projected maximum use temperature due to concerns about the longterm microstructural instability of heat resistant steels.

Martensitic Steel Development



- 1950's to date Low alloy creep resisting steels
- 2¼CrMo; CrMoV
 - Ferritic structure, limited carbide strengthening
 - Applications up to about 540 570°C (maximum)
- 1980's development P91 or "Modified 9Cr-1Mo" steel
- Introduced from early 1990's onwards
- Coal plant boiler headers and drums (UK first), steam pipework and HRSG applications worldwide
 - Martensitic structure
 - Fine scale lath structure for increased creep strength
 - Carbide precipitate chains on lath boundaries
 - Vanadium modified to add finer-scale network of VN/MX precipitates
 - Applications generally up to about 580°C (or higher if at low stress)

1990 - 2000 - P92 steel (and others MARBN, <u>CPJ-7</u>, etc.)

- For example, replace Mo in P91 with W in P92; incorporate B: Creep strength increase in P92 compared to P91
- Applications e.g., 600°C main steam, 620°C hot steam reheat

Computational / Experimental Alloy Design and Process Development Approach



- Model & design alloys using computational thermodynamics software (ThermoCalc) to develop the phases required for creep strength & to maintain the martensitic nature of the steel.
- Formulate, melt & cast alloy heats for each composition using best melting practice for alloy formulation.
- Homogenize each alloy according to its own <u>computationally</u> <u>optimized</u> heat treatment schedule developed from thermodynamic (ThermoCalc) & kinetic (DICTRA) modeling approach.
- Fabricate alloys into plate using standard hot forging & rolling operations.
- Develop desired microstructure features & steel strength through normalizing & tempering heat treatments.
- Assess creep & tensile properties against COST alloys (turbine) and P91 / P92 (boiler).

General Technical Approach



- Understand basic high temperature strengthening mechanisms & how to preserve strengthening effect through microstructural control.
- Achieve balance between the following competing effects:
 - Necessary <u>C</u>, <u>V</u>, <u>Nb</u>, (and/or <u>Ta</u>) and <u>N</u> to generate MX (M: is metal; X: is C/N), thereby, slowing down dislocation movement in the matrix during creep.
 - Balanced amount of <u>Mo</u> and <u>W</u> for solution & precipitation hardening by $M_{23}C_6$ (and very small Laves phase).
 - Addition of <u>Co</u>, <u>Cu</u>, <u>Mn</u>, and/or <u>C</u> to suppress δ-ferrite & to provide additional precipitate strengthening (<u>Cu</u>) & oxidation resistance (<u>Mn</u>).
 - Addition of <u>B</u> to stabilize $M_{23}C_6$ precipitates, and thus, help to stabilize the prior austenite grain and sub-grain structures.
 - Higher level of <u>C</u>r for oxidation resistance (e.g., must be balanced because Cr additions significantly greater than 8.5 to 9% reduce creep strength).
 - Addition of <u>S</u>i and/or <u>RE</u> elements to improve oxidation resistance.

Agamennone et. al. Acta Mater. (2006), Knezevic et al. Mater. Sci. Eng. A. (2008), Wang et al. Mater. Sci. Eng. A. (2009), Yin & Jung, J. Mater. Pro. Technol. (2009), and Chilukuru et al. Mater Sci. Eng. A. (2009).

Microstructural Hierarchy of 9-12% Chromium Containing Steels





- 1. Prior austenite grain with associated grain boundaries.
- 2. Packet boundaries
- 3. Block boundaries
- 4. Lath boundaries
- 5. M₂₃C₆ carbides to stabilize lath, block, packet, and PAG boundaries
- 6. MX carbides to provide obstacles to dislocation motion
- 7. Dislocations

The premature breakdown of any one of these microstructural features will destabilize the entire alloy, and lead to ever increasing creep rate over time. The goal of alloy design is to slow down the destabilization of these features starting with the MX and $M_{23}C_6$ particles.

F. Abe, "Metallurgy for Long-term Stabilization of Ferritic Steels for Thick Section Boiler Components In USC Power Plants at 650°C," Proceedings of the 8th Liege Conference, (2006), pp. 965-980.

Microstructural Stability of 9-12%Cr Steels



USC Materials Development Experience in Precipitate Instability



Many competing effects occur in heat resistant steels of the 9% Cr variety. Past experience has shown that the instability of any of the following, Z-phase, Laves, MX and/or $M_{23}C_6$, can cause an unexpected decrease in rupture stress as a function of time. The goal of alloy design is to slow down the destabilization of these features starting with the MX and $M_{23}C_6$ particles.

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Summary of Major Commercial 9%-12% Cr Steels Versus CPJ-7 Alloys



Chemistry]													
Material	С	Mn	Si	Ni	Cr	Мо	V	Nb	N	W	В	Со	Fe	Та
COST FB2	0.13	0.30	0.08	0.05	9.30	1.50	0.20	0.05	0.026		0.010	1.00	Bal	
COST E	0.12	0.45	0.10	0.74	10.40	1.10	0.18	0.045	0.05	1.00			Bal	
COST B2	0.18	0.06	0.10	0.09	9.28	1.54	0.29	0.06	0.02		0.010		Bal	
P91	0.10	0.45	0.35	0.20	8.50	0.90	0.21	0.07	0.045				Bal	
P92	0.09	0.42	0.30	0.15	9.10	0.40	0.20	0.06	0.045	1.75	0.002	0.01	Bal	
MARBN	0.15	0.51	0.24	0.18	9.10	0.10	0.20	0.06	0.023	2.90	0.012	2.95	Bal	
CPJ-7	0.15	0.41	0.09	0.27	9.83	1.26	0.21	0.056	0.020	0.48	0.0100	1.48	Bal	0.28
CPJ-7B	0.15	0.29	0.15	0.22	9.81	1.46	0.20	0.059	0.025	0.43	0.0078	1.53	Bal	0.20
CPJ-7C	0.16	0.47	0.11	0.22	9.95	1.34	0.19	0.061	0.022	0.49	0.0086	1.59	Bal	0.20
CPJ-7D	0.16	0.43	0.10	0.22	10.12	1.31	0.21	0.054	0.024	0.53	0.0083	1.56	Bal	0.24
CPJ-7E	0.15	0.42	0.12	0.21	9.99	1.35	0.20	0.049	0.022	0.53	0.0087	1.51	Bal	0.28

The following elements were also found in the CPJ-7 Alloys: Ti (<0.004%), Al (<0.02%), P (<0.003%), Cu (<0.003%), O (<36 ppm), and S (<58 ppm).

CPJ 9% Cr Martensitic Steel Design Matrix



ID	Mn	Si	Cr	Ni	Со	Мо	W	Nb	Ti	Al	Fe	Cu	Та	Hf	Re	V	С	0	N	Р	S	В
#	Wt%	Wt%	Wt%	Wt%	Wt%	Wt%	Wt%	Wt%	Wt%	Wt%		Wt%	Wt%	Wt%	Wt%	Wt%	Wt%	ppm	ppm	Wt%	ppm	ppm
1	0.480	0.24	10.08	0.070	2.93	0.01	0.025	0.053	0.095	0.013	BAL	0.003	0.003			0.21	0.10	92	111	0.001	60	100
2	0.490	0.26	10.08	0.060	0.01	1.53	0.010	0.057	0.097	0.013	BAL	0.003	0.001			0.21	0.10	72	96	0.001	60	100
3	0.508	0.261	10.111	0.080	2.931	0.010	0.037	0.054	0.010	0.017	BAL	0.003	0.145			0.206	0.18	30	161	0.001	60	100
4.1	0.406	0.087	9.833	0.270	1.479	1.262	0.482	0.056	0.004	0.016	BAL	0.003	0.279			0.209	0.15	36	200	0.001	50	100
4.2	0.288	0.150	9.812	0.217	1.529	1.464	0.428	0.059	0.004	0.005	BAL	0.003	0.202			0.204	0.15	29	252	0.003	58	78
4.3	0.473	0.111	9.953	0.224	1.588	1.342	0.493	0.061	0.004	0.009	BAL	0.003	0.198			0.194	0.16	34	222	0.003	55	86
4.4	0.430	0.101	10.121	0.215	1.557	1.312	0.526	0.054	0.004	0.010	BAL	0.003	0.244			0.212	0.16	36	245	0.001	58	83
4.5	0.421	0.117	9.993	0.206	1.505	1.325	0.530	0.049	0.004	0.014	BAL	0.003	0.280			0.199	0.15	32	221	0.001	58	87
4.6	0.430	0.106	10.034	0.214	1.585	1.365	0.508	0.058	0.004	0.005	BAL	0.031	0.155			0.219	0.15	64	278	0.003	59	93
4.7	0.405	0.065	9.963	0.310	1.467	1.263	0.484	0.056	0.001	0.013	BAL	0.028	0.377			0.202	0.15	66	222	0.001	38	92
4.8	0.393	0.055	9.986	0.310	1.480	1.262	0.480	0.056	0.001	0.012	BAL	0.026	0.310			0.206	0.15	73	208	0.001	42	90
4.9	0.406	0.071	9.969	0.310	1.472	1.262	0.491	0.056	0.003	0.014	BAL	0.027	0.368			0.207	0.15	67	176	0.001	38	89
4.10	0.471	0.096	9.847	0.270	1.458	1.261	0.484	0.057	0.002	0.011	BAL	0.294	0.280			0.208	0.15	32	239	0.001	56	104
4.11	0.414	0.078	9.875	0.270	1.467	1.268	0.472	0.056	0.001	0.017	BAL	0.006	0.269		0.115	0.202	0.15	32	238	0.001	58	102
5	0.415	0.101	10.639	0.280	2.933	0.504	0.513	0.056	0.003	0.013	BAL	0.033	0.266			0.205	0.15	50	341	0.001	60	89
6	0.403	0.084	10.629	0.270	2.937	0.505	0.517	0.055	0.010	0.012	BAL	0.031	0.003			0.203	0.14	75	200	0.001	64	87
7	0.410	0.101	10.176	0.227	1.586	1.347	0.596	0.051	0.004	0.005	BAL	0.042	0.005	0.160		0.213	0.15	47	215	0.002	59	97
8	0.422	0.099	10.618	0.234	4.673	0.539	0.479	0.054	0.004	0.005	BAL	0.043	0.177			0.190	0.15	28	296	0.002	67	97
9	0.452	0.101	10.527	0.269	6.158	0.527	0.508	0.055	0.004	0.011	BAL	0.037	0.260			0.237	0.15	28	328	0.002	67	88
10	0.443	0.013	10.740	0.260	8.226	0.524	0.498	0.053	0.004	0.005	BAL	0.037	0.185			0.197	0.15	23	379	0.001	75	95
11	0.413	0.084	9.875	0.260	0.012	1.262	0.479	0.056	0.003	0.016	BAL	0.002	0.266			0.205	0.15	43	244	0.001	50	83
12	0.409	0.057	9.975	0.310	2.949	1.267	0.458	0.062	0.001	0.013	BAL	0.026	0.355			0.208	0.15	72	221	0.001	39	75
13	0.403	0.054	9.969	0.310	0.736	1.269	0.468	0.061	0.002	0.013	BAL	0.027	0.361			0.210	0.15	74	238	0.001	36	75
14	0.419	0.088	9.893	0.270	1.460	0.502	0.515	0.054	0.002	0.011	BAL	0.295	0.233			0.206	0.15	50	221	0.001	50	113
15	0.411	0.085	9.866	0.270	1.466	0.506	0.511	0.055	0.001	0.017	BAL	0.003	0.274		0.100	0.205	0.15	27	236	0.001	59	106
16	0.412	0.078	9.111	0.270	1.474	1.370	0.476	0.056	0.002	0.014	BAL	0.033	0.346			0.206	0.15	28	226	0.001	59	85
17	0.510	0.229	9.085	0.190	2.942	0.100	2.914	0.062	0.001	0.014	BAL	0.003	0.010			0.202	0.15	27	193	0.001	59	104
18	0.511	0.257	9.085	0.180	2.926	0.101	2.894	0.065	0.001	0.014	BAL	0.003	0.003			0.208	0.15	21	230	0.001	67	121
19	0.512	0.240	9.080	0.180	2.928	0.100	2.897	0.063	0.001	0.016	BAL	0.032	0.349			0.203	0.15	22	222	0.001	60	101



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NETL 9% Cr Martensitic-Ferritic Steel Screening Creep Tests at 25 ksi



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Summary of Tensile Mechanical Behavior of CPJ-7 Alloys





Larson Miller Parameter for COST E Steel & Wrought CPJ-7 Steel



Larson-Miller Parameter plot for COST E at temperatures from 1050°F (565.5°C) to 1200°F (648.9°C). CPJ-7 testing performed at 650°C only.



Additional creep tests are being run to define LMP & isothermal curves more completely.

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 (Left) HRTEM micrograph showing fine coherent MX-type precipitates (indicated by arrows) in the martensitic matrix of as-received CPJ-7.
(Right) A magnified view of the lower precipitate in the left panel. The precipitate is located at the center of two high-strain (white) regions that result from lattice mismatch between the precipitate and the matrix and/or a possible interaction with the strain field of a dislocation (not visible in this orientation).

Wrought CPJ-7 vs Current Materials Used for Steam Turbine Rotors in Power Plants





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NETL Examined the HT Creep Potential of MARBN



- NETL have examined the potential of W-bearing 9% Cr martensiticferritic steels similar to MARBN (MARtensitic 9Cr steel strengthened by Boron and MX Nitrides).
- ❑ Limited mechanical testing: tensile behavior vs. temperature (up to 650°C) and creep screening at 650°C (25, 22.5 & 20 ksi stress levels)
- □ NETL made three variants:
 - MARBN 1A based on best chemistry and heat treatment (no homogenization but normalized) information available in literature plus NETL tempering conditions
 - MARBN 1B based on best chemistry information available in the literature (same as MARBN 1A), and NETL homogenization step plus standard normalization treatment and tempering conditions
 - MARBN 2 based on some changes to the basic MARBN 1 chemistry but using NETL homogenization step plus standard normalization treatment and tempering conditions

MARBN Steel Variants: Tensile Behavior





MARBN steels possess similar tensile behavior to the CPJ-7 martensitic-ferritic steels. This most probably arises from the quality control and reproducibility of the manufacturing process used at NETL.

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Preliminary Creep Potential for MARBNbased Steel (NETL conditions)



Alloy / Test	Creep Life (h)								
Conditions	25 ksi	22.5 ksi	20 ksi						
MARBN 1A	<u>2,275</u>	<u>4,587</u>	7,694						
MARBN 1B	1,833	3,443	<u>8,201</u>						
MARBN 2	1,030	1,387	2,458						

Preliminary Observations

- Performance of MARBN 1A is very good, better than CPJ-7 at these stress levels.
- MARBN 1B seems to perform very well at 138 MPa. This is similar to cast CPJ-7 at this stress level.
- MARBN 2 does not perform as well as MARBN 1 or CPJ-7.

CPJ7 vs State-of-Art Experimental Boiler Steel MARBN



NIMS 9Cr steel : MARBN

MARBN : **MAR**tensitic 9Cr steel strengthened by **B**oron and MX **N**itrides

MARBN : 9Cr-3W-3Co-VNb, 120 - 150 ppm B & 60 - 90 ppm N P92 : 9Cr-0.5Mo-1.8W-VNb, 20 ppm B & 500 ppm N



Fireside Corrosion Potential – Alloys & Ash Compositions (wt%)



Alloy	Fe	Cr	Ni	Со	Мо	С	Si	Ti	Al	Mn	V	Nb+Ta	Cu	Other
T92	Bal	9.08	0.25	0.01	0.45	0.081	0.09		0.01	0.40	0.21	0.07		1.80 W
CPJ 7	Bal	9.83	0.27	1.48	1.26	0.15	0.09	0.004	< 0.02	0.41	0.21	0.336	0.03	0.48

ID	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	Na ₂ SO ₄	K ₂ SO ₄
SCM	0	0	25	37.5	37.5
S80	10	10	20	30	30
S60	20	20	15	22.5	22.5
S40	30	30	10	15	15
S20	40	40	5	7.5	7.5

Ash Compositions

- Maintain 3:1 ratio of (Na,K)₂SO₄:Fe₂O₃ as found in lowest melting point alkali iron trisulfates.
- Different alkali sulfate fluxes to alloy surfaces.



Fireside Corrosion Potential -Metal Loss Results (240 h)







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High Temperature Air Oxidation – CPJ-7 Oxidation Potential







Wrought NETL CPJ-7 Advanced 9% Cr Steel



- Identified promising chemistry for ferritic-martensitic steel, CPJ-7, through control of minor alloying additions (C, Cu, Ta) and B/N levels.
- Developed manufacturing approach to consistently produce CPJ-7.
- Utilized NETL homogenization step in conjunction with thermomechanical processing to set and stabilize microstructure.
- Tested CPJ-7 chemistry robustness by varying select combinations of alloying additions: Mo_(eqv); C + N level; B level – producing and testing four additional CPJ-7 heats.
- □ Assessed other minor element additions and extent of those additions on tensile and creep strength of CPJ-7 base alloy.
- Patent awarded: Hawk, Jablonski & Cowen, Creep Resistant High Temperature Martensitic Steel, US 9,181,597 B1, November 10, 2015.

Brief Description of Cast NETL CPJ-7 Advanced Martensitic Steel



- Previous research identified NETL martensitic-ferritic steel CPJ-7. A <u>wrought</u> product was manufactured.
- NETL <u>wrought</u> CPJ-7 steel exhibited superior creep strength compared to commercially designed, thermo-mechanically processed and heat treated 9% Cr martensitic steels used for airfoils, rotors, and other wrought components in a steam turbine as well as piping and other thermo-mechanically processed components in the combustion boiler.
- NETL applied same alloy design rationale to develop <u>cast</u> martensitic 9% Cr steel. Subsequent alloy homogenization using NETL algorithmic approach with subsequent martensitic steel heat treatment produced <u>cast</u> version of CPJ-7 superior to any existing commercially available <u>cast</u> 9% Cr martensitic steel or derivatives.

Wrought vs. Cast Manufacturing



Wrought Manufacturing Steps:

- 1. Alloy Design
- 2. Melt Processing
- 3. Homogenization
 - Improve chemical uniformity within the matrix structure
- 4. Thermo-mechanical Processing
 - Physical manipulation of the grain structure for mechanical property design & refinement
 - More homogeneous "physical" structure – i.e., a more consistent & uniform grain size
- 5. Heat Treatment for Strength

Cast Manufacturing Steps:

- 1. Alloy Design
- 2. Melt Processing
- 3. Homogenization
 - Improve chemical uniformity within the matrix structure
- 4. Heat Treatment for Strength
- Major difference is no manipulation of the "physical" grain structure of the resulting solid body.
- Limited ability to develop strength in the solid body except through alloy design & heat treatment.

Martensitic Steel Ingot Casting



Large-scale Steel Casting for USC 650°C Power Plants:

Heats of CPJ-7 were formulated and cast utilizing NETL's "enhanced slow cooling" methodology. The mold was submerged in loose sand to help contain the heat of the molten steel, and thereby, slow the cooling rate substantially in order to better simulate the slow cooling conditions of a thick wall, full-size steam turbine casings. The fully heat treated ingot was then bisected along the diameter. The halves were then sectioned into 0.4" thick slabs from which 0.4" square bars were cut. From these squares round tensile bars were subsequently machined into traditional tensile/creep specimens.



Cast 9% Cr Martensitic Steel Chemistry



- This new *cast* 9% Cr martensitic steel has a unique chemistry, • alloy design philosophy, and microstructural control (i.e., computationally based homogenization heat treatment schedule) unlike any other alloy in it's class.
- Nominal/preferred alloy chemistry: ۲

	С	Mn	Si	Ni	Cr	Мо	V	Nb	N	W	В	Со	Fe	Та
CPJ-7	0.15	0.40	0.10	0.30	9.75	1.25	0.20	0.06	0.020	0.50	0.0100	1.50	Bal	0.20

- Alloy design philosophy: \bullet
 - Slow down the destabilization of the various grain boundary & matrix strengthening features such as MX and $M_{23}C_6$ particles.
 - Avoid and/or postpone the formation of unwanted phases such as the Z-phase and Laves phase.
- Homogenization: ٠
 - Induce complete chemical uniformity on the micro-scale to avoid "over rich" or "over lean" regions that could promote deleterious phase formation, thereby achieving long-term alloy stability.



Cast 9% Cr Martensitic Steel – Manufacturing Proof of Principle



- The preferred chemistry for cast CPJ-7, 9% Cr martensitic steel, was used to manufacture three heats. (No attempt was made to optimize the casting process at this time.)
- After homogenization, the cast CPJ-7 9% Cr steel ingot was heat • treated in the following manner:
 - 1150°C/30 min/AC + 700°C/1 hour/AC
- Screening tensile tests were performed from material that solidified in an <u>equiaxed</u> manner (i.e., center of the casting) as well as from material that solidified in a *columnar* manner (i.e., exterior surface region of casting).
- Creep tests from 30 ksi to 17.5 ksi and 650°C have been performed to assess the extent of creep capability relative to commercial cast steels used in power plants, (e.g., COST CB2).
- Mechanical performance looks *very good* with cast CPJ-7 showing • outstanding mechanical performance for a casting, and similar to wrought CPJ-7.



Proof of Principle Detailed Tensile Mechanicals





Additional heat of cast CPJ-7 are undergoing tensile and creep testing.



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Proof of Principle Creep Screening Tests





Proof of Principle Creep Behavior Compared To CB2



	CB2	CB2	Cast	CPJ-7	Average LMP		
Alloy/Stress	Failure Time	LMP	Time (LC)	Time (UC)	LMP (LC)	LMP (UC)	
30.0				1,626		46.88	
27.5				$2,377\pm678$		47.14	
25.0	27.5^	43.95^	861 ± 30		46.42		
22.5	87.5^	44.78^	1,493 ± 50	5,580	46.82	47.78	
20.0	278.5^	45.61^	3,496 ± 308	8,396*	47.43	48.07*	
17.4	$282\pm257^{\#}$	45.43#	$5,528\pm460$		47.76		
14.5	$2,716 \pm 1,018^{\#}$	47.23#					
12.3	15,943 ± 2,802#	48.53#					

For CB2

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^ Extrapolated data from mean curve# Data from two tests

For CPJ-7 * Specimen still in test (~ 1.15% creep strain)

Comparison Between Wrought & Cast CPJ-7



Additional wrought heat manufactured and tested as well as additional tests from prior heats. Another casting has been made with tensile specimens in the queue for testing.



Wrought & Cast CPJ-7 Compared to Representative Commercial Steels



Larson-Miller Parameter plot for COST E & cast CB2 at temperatures from 1050°F (565.5°C) to 1200°F (648.9°C). CPJ-7 (cast (x) & wrought (+)) testing performed at 650°C only.

Summary & Planned Next Steps



- Continue short-term creep testing through fall of 2016 & into 2017.
- Optimize casting process to reduce porosity & produce "sounder" castings (i.e., through the use of gating system, risers, filters, etc.).
- Produce wrought plate & cast blocks suitable for welding studies. Explore alternative joining technologies.
- Consider making large CPJ-7 casting via air induction melting process. Assess limitations of approach relative to baseline data.
- Assess toughness & fracture energy, followed by selected fatigue screening tests at room temperature for CPJ-7 (wrought first, then castings).
- Initiate longer term creep testing (wrought & cast).
- Submitted new patent application to encompass claims on castings: Hawk, Jablonski & Cowen, *Creep Resistant High Temperature Martensitic Steel*, continuation of and claims priority to 13/868,139 (04/23/2013) & US 9,181,597 B1 (11/10/2015) by the same inventors, the entirety of which is incorporated by reference (9/30/2015).





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