

Nickel-Base Superalloys for Advanced Power Systems – An Alloy Producer’s Perspective

John J. de Barbadillo
Manager, Product and Process Development
Special Metals Corporation
Huntington, WV, USA
email jdebarba@specialmetals.com

Brian A. Baker
Application Engineer
Special Metals Corporation
Huntington, WV, USA
email bbaker@specialmetals.com

Ronald D. Gollihue
Welding Specialist
Special Metals Corporation
Huntington, WV, USA
email rgollihue@specialmetals.com



John de Barbadillo received his BS, MS and PhD degrees from Lehigh University. He has been employed by Inco Ltd R&D Center in Suffern NY and then at the Inco Alloys/Special Metals plant in Huntington WV. He has worked on a wide range of nickel alloy technologies with recent specialization in advanced remelting processes. Dr. de Barbadillo is a Fellow of ASM and currently holds the position of Manager, Product and Process Development.



Brian Baker is a Product Development and Application Engineer with 24 years of experience working in the areas of high temperature corrosion and product development with Special Metals. He has numerous publications, patents and associated new products and methods.



Ronald D. Gollihue is an American Welding Society Certified Welding Inspector and Certified Welding Educator. He has been in the welding industry for 25 years. For the past 14 years he has been a Welding Project Specialist for Special Metals Research and Technology group overseeing the day-to-day functions of the Welding Development Laboratory in Huntington WV.

ABSTRACT

There is a broad-based, world-wide effort to develop and qualify nickel-base superalloys for service in advanced electrical power generation systems. Although superalloys have been used for about seventy

years in aircraft gas turbine engines, the product forms, sizes and mechanical and corrosion properties needed for power plants necessitated extensive redevelopment of these alloys. Several alloys have been evaluated and found suitable for use in various components of advanced boiler and turbine systems. This paper focuses on one alloy, INCONEL® alloy 740H®, that is currently the only age-hardened superalloy approved for use in welded and creep-limited pressure applications by the ASME boiler code. The microstructure stability, creep and corrosion properties have been widely studied and reported in the literature. Primary product forms such as tube and pipe have been manufactured and characterized sufficiently to establish that their properties meet code requirements. The current manufacturing demonstration focus is on fabricating bends, flanges, “tees”, “wyes” and other components needed to construct a power plant. Extensive welding studies are also underway, including a demonstration of stress relaxation cracking resistance and thick-section dissimilar metal welding capability. This paper reviews the reason to consider age-hardened alloys, the status of recent development activity and the question of whether the alloy is ready for use in power plants.

® Registered trademarks of Special Metals Corporation

INTRODUCTION

Nickel-iron-chromium base materials such as alloy 800HT (UNS N-08811) have been used in welded service for containment of fluids and gases in creep-limited applications in the chemical process and fossil and nuclear power generation industries for many years. These alloys are primarily solid solution strengthened and are well characterized, but they have relatively low strength. Subsequently, higher strength alloys that contain cobalt and higher levels of carbon and refractory metals were developed. Alloy 617 (UNS N06617) that was introduced in the 1970s, has been intensively studied for service at temperatures above 650°C (1202°F). This alloy has been used in sheet and plate form primarily for combustors and ducting in aircraft and industrial gas turbines and for chemical process vessels. Although it has been used successfully in those applications, some issues relevant to its use in power plants such as microstructure stability and stress relaxation cracking of welds remain. In addition, the temperature and pressure design goals for the Advanced Ultra-supercritical (AUSC) coal-fired power boiler would require very thick walls for tube and pipe made from alloy 617. Because the alloy is very expensive compared to stainless steel, there is a strong motivation for use of even higher strength alloys. This material gap was recognized at the outset of the European Thermie program in the late 1990's. The project planners then began to consider the possibility of using age hardened alloys.

The addition of aluminum, titanium, and niobium to nickel-base alloys can generate an intense hardening reaction when the material is “aged” for a period of time at an intermediate temperature. The hardening reaction involves the precipitation of an intermetallic compound of formula Ni_3X where X may be a mixture of Al, Ti and Nb. The precipitate's cubic crystal structure is designated gamma prime (γ'). Alloys of this type are commonly called “superalloys” and they have essentially enabled the success of the aircraft turbine engine. Over the years these alloys have been customized for the specific requirements of various engine components such as rotors, airfoils and casing. One class of superalloy that contains 15-25 volume percent (vol. %) γ' , is used in sheet form for welded and formed casing, ducts and combustors has general characteristics that make them candidates for use in tube and pipe for power plants. Several distinct alloys have emerged from that original work as potentially suitable for use in power plants. In particular, alloy 740H (UNS N07740) was specifically developed and characterized under the European and US A-USC programs. The alloy meets all of the original design requirements for A-USC service. Its properties have been extensively documented [Baker 2013, Shingledecker 2013]. The data generated, primarily from the US program, served as the base for ASME Code Case 2702 [ASME 2011].

Age-hardened alloys have characteristics that are very desirable for power plant use. They are relatively soft and formable in the solution treated condition. In this condition they can be rolled and drawn to form sheet and tube, and tube can be cold formed by bending. Subsequently the components are again solution treated and aged. Direct aging of cold formed parts is restricted due to a loss of creep strength. Alloys of this type can be welded successfully although strict protocols must be followed and high energy processes cannot be used. Concerns about use of age-hardened alloys in this application include long time microstructure stability (> 100,000 h. at temperature), impact toughness, creep, creep-fatigue, over temperature damage tolerance and strain-aging and stress relaxation cracking of welds. Understanding of the issues is being developed through continued laboratory work, component manufacture, and in-plant

test loops and pilot plants. The following discussion presents recent developments and remaining challenges using alloy 740H as the example. These findings can be applied to other alloys of this class as they are developed and qualified.

RESULTS AND DISCUSSION

Mechanical Properties

The nominal composition of alloys 617, 740H and 800HT are shown in Table I. Note that alloys 617 and 800 do contain additions of Al and Ti and can form up to 5 vol. % of γ' during long-time exposure; however, the effect on yield strength is small and hence they are not considered age-hardened alloys. They are normally used in the solution annealed condition. In contrast, 740H an alloy that is single phase after solution anneal and rapid cooling, can precipitate 15-20 vol. % γ' after age-hardening. The preferred heat-treatment is solution anneal 1100°C (2010°F) followed by aging at 760-816°C (1400-1500°F). Note that the molybdenum in 617 and cobalt in 617 and 740H also contribute significantly to solid solution strengthening.

Table I. Nominal composition of Alloys 617, 740H and 800HT. Weight Percent. R = Residual

Alloy	Ni	Fe	Cr	Co	Mo	Al	Ti	Nb	C
800HT	31	Bal	20	R	R	0.5	0.5	R	0.08
617	Bal	0.5	22	12.5	9	1.15	0.3	R	0.08
740H	Bal	0.25	24.5	20	0.25	1.35	1.35	1.5	0.03

The ambient temperature tensile and impact properties of alloy 740H in both the solution annealed (SA) and solution annealed and age hardened (SA+A) conditions are shown in Table II. This data was obtained on a range of tube and pipe sizes and manufacturing methods. For example tube was either cold drawn or pilgered prior to heat treatment. Pipe was produced by hot extrusion and direct heat treatment. Tube was heat treated by either continuous or batch annealing. This indicates that the alloy is capable of meeting ASME Code requirements using standard mill equipment. There is no impact toughness requirement in the ASME code; however, fabricators have a concern if toughness is too low. This property will be discussed in greater detail under microstructure stability. Solution annealed properties are of interest for fabricators who need to do cold forming or bending. Elevated temperature tensile properties are shown in Table III.

Table II. Room temperature tensile and impact properties of alloy 740H

Form	Size (OD x ID) mm, in.	Heat Treatment	No. Tests	YS, ksi, MPa	TS, ksi, MPa	El., %	RA, %	CVN, J/cm ² , Ft. lb
ASME Min.	All	SA + A		620 (90)	1035 (150)	20		
Tube	21.3 (0.84) x 2.78 (0.11)	SA	2	512 (73)	943 (137)	55.9		
	38.1 (1.5) x 7 (0.28)	SA	3	394 (57)	784 (114)	56.3		
	38 (1.5) x 8.8 (0.35)	SA	24	503 (73)	944 (137)	51.7		65.1 (88.3)
	50.8 (2) x 8 (0.32)	SA	6	405 (59)	824 (120)	56.2		83.7 (114)
	21.3 (0.84) x 2.78 (0.11)	SA + A	2	746 (108)	1163 (169)	42.8		
	38.1 (1.5) x 7 (0.28)	SA + A	3	769 (111)	1118 (162)	33.9		
	38 (1.5) x 8.8 (0.35)	SA + A	21	761 (110)	1164 (169)	37.0		33.5 (45.4)
	42.2 (1.66) x 10 (0.39)	SA + A	2	731 (106)	1158 (168)	36.3		34.4 (46.6)
	42.2 (1.66) x 11 (0.43)	SA + A	2	736 (107)	1153 (167)	37.0		32 (43.4)
	50 (1.96) x 10 (0.39)	SA + A	1	747 (108)	1172 (170)	37.5		
	50 (1.96) x 8 (0.31)	SA + A	6	766 (111)	1174 (170)	35.8		29.4 (39.9)
Pipe	358 (14.1) x 88 (3.46)	SA + A	4	728 (106)	1094 (159)	31.8	32.4	30.2 (41)
	324 (12.8) x 22.5 (0.88)	SA + A	2	741 (107)	1083 (157)	33.6	36.2	
Bar	76.2 (3)	SA + A		736 (107)	1147 (166)	36.5		

Table III Elevated Temperature tensile properties of alloy 740H. Material in SA + A Condition

Product	Size (OD x Wall), mm (In.)	Temp., °C (°F)	No. Tests	YS, MPa (ksi)	TS, MPa (ksi)	El%
Tube	38 (1.496) x 8.8 (0.346)	650 (1202)	2	638 (92)	985 (142)	25.5
		700 (1292)	1	682 (99)	964 (140)	23.2
		750 (1382)	2	621 (90)	845 (122)	17.4
	50 (1.969) x 10 (0.394)	650 (1202)	1	627 (91)	978 (142)	28.5
		700 (1292)	1	670 (97)	932 (135)	18.5
		750 (1382)	1	649 (94)	836 (121)	15.0
50.8 (2) x 8 (0.315)	650 (1202)	2	610 (88)	950 (138)	27.0	
	700 (1292)	2	637 (92)	906 (131)	16.2	
	750 (1382)	2	610 (89)	794 (115)	12.5	
Pipe - 740H	358.2 (14.1) x 76.6 (3.02)	650 (1202)	1	600 (87)	924 (134)	29.8
		700 (1292)	1	572 (83)	889 (129)	15.9
		750 (1382)	1	573 (83)	800 (116)	22.5
	323.8 (12.784) x 22.5 (0.886)	650 (1202)	2	590 (86)	876 (127)	30.7
Bar - 740	76.2 (3)	650 (1202)	1	596 (86)	944 (137)	33.0
		700 (1292)	1	626 (91)	982 (142)	19.5
		750 (1382)	1	623 (90)	827 (120)	28.0

Alloy 740H was optimized to balance creep-strength, weldability and corrosion resistance. The creep-rupture properties have been determined on a wide variety of product forms with a range of composition. This data is shown in parametric form in Figure 1. The solid line represents the data submitted to ASME for the code case. Most of this data has been developed in testing at Oak Ridge National Lab under sponsorship of the US A-USC Consortium. The data extend to 50,000 h with some tests continuing that should exceed 100,000 h. The data is reported and analyzed by Shingledecker [2012]. Figure 2 shows the ASME limiting design stress for alloys 617, 740H and 800HT. Alloy 740H shows a significant strength advantage in the temperature range of interest for AUSC. At temperatures above 800°C (1472°F) alloy 740H loses its strength advantage because γ coarsens and the volume fraction diminishes. The actual γ solvus is 973°C (1783°F). The creep rupture life data for mill product forms do tend to fall below that of the qualification data, but within the conventional 20% range. It is not clear at this point whether this is a testing issue (different test location, specimen geometry, etc.), composition issue or factors related to the structure of commercial product or the manufacturing process itself is not yet optimized..

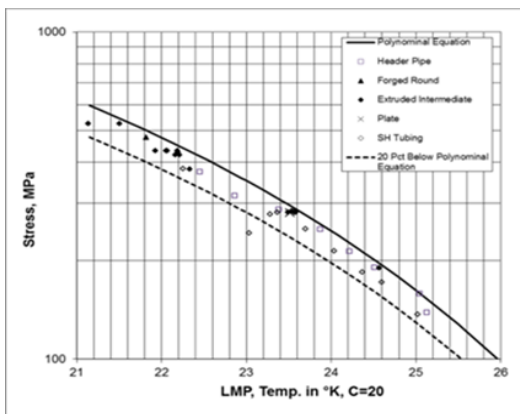


Figure 1. Parametric rupture life data for 740H.

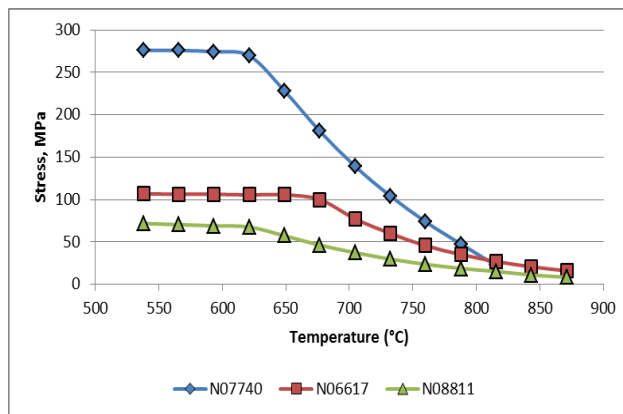


Figure 2. ASME allowable design stresses

Microstructure and stability

Many superalloys undergo microstructural changes if they are held for long periods of time at elevated temperatures. These changes may include formation of complex carbides, growth of γ and the formation of a variety of topological close-packed (TCP) phases including Laves and sigma. These phases may form undesirable morphologies that reduce ductility and toughness. In the application of superalloys for power plant use it is necessary to demonstrate that they will be stable for the design lifetime of the plant.

To evaluate the microstructural stability of 740H, material obtained from commercially produced pipe was exposed stress-free for times up to 10,000 h at 700°C (1292°F), 750°C (1382°F) and 800°C (1472°F). Longitudinal Charpy V-notch impact tests were conducted and the microstructure was analyzed in detail. The results of this investigation that were previously reported [deBarbadillo 2014a], are summarized below.

A representative scanning electron micrograph (SEM) of material in the as heat treated condition is shown in Figure 3a. The grain interiors show a uniform fine γ precipitate. The grain boundaries are almost completely covered with $M_{23}C_6$ and coarse γ . Material exposed for 5,000 h at 750°C (1382°F) is shown in Figure 3b. Note that the γ has grown significantly, but it continues to maintain a generally cubic morphology. Larger chunky γ decorates the grain boundaries. Most of the remaining grain boundary area is covered by $M_{23}C_6$ carbide. Detailed phase identification was performed by Xie and coworkers [Zhao 2013]. He found no evidence of η or G phase. Sigma phase is predicted by Thermo-Calc to form at 650°C (1202°F) in this alloy at very long exposure times, but to date, it has not been reported. Creep deformation sometimes accelerates microstructural changes by providing enhanced diffusion and nucleation sites. Based on limited observations this does not seem to be the case for 740H. Figure 3c shows the structure near the fracture surface of a specimen tested at 750°C (1382°F) with an initial applied stress of 280 MPa (40.6 ksi) that broke in 1087.4 h. The structure is similar to the starting structure, but a chain of grain boundary voids is visible. Test bar fracture occurs by linking of these voids. There was no sign of a precipitate-free zone adjacent to the grain boundary although this feature has been observed in welds of 740H.

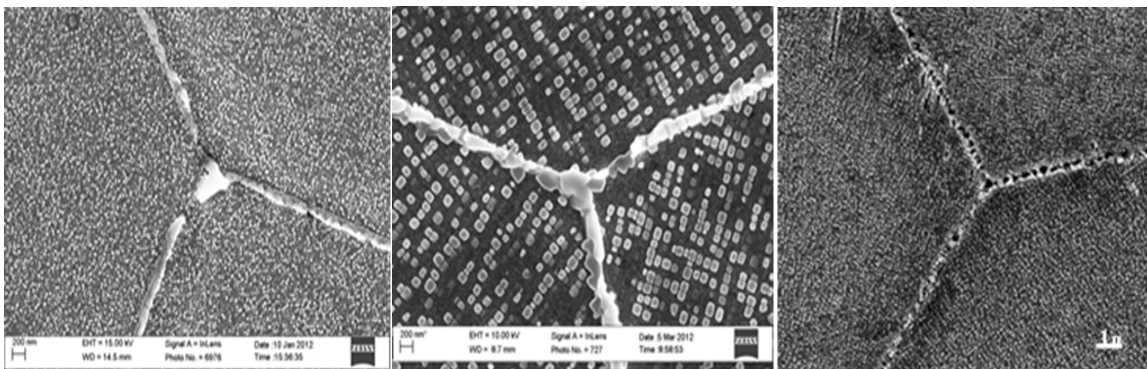
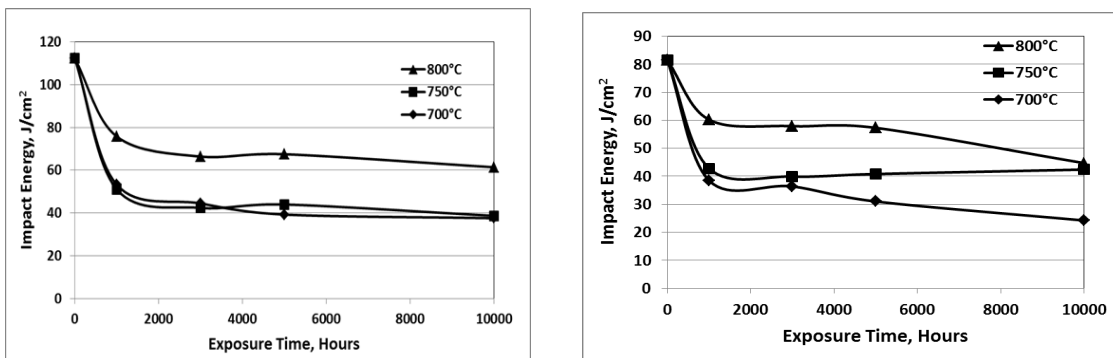


Figure 3. SEM Micrographs: a) As heat treated, b) Stress-free exposure for 5000 h at 750°C (1382°F), c) Gauge section of creep-rupture specimen failed at 1087.4 h at 750°C (1382°F) and 280 MPa (40.6 ksi).

Impact toughness was determined on both tube and pipe samples. The results are shown in Fig 4a and 4b. Toughness drops off during the first 1000 hours. This is attributed to precipitation of additional γ and the formation of a thicker grain boundary carbide network. After about 1000 h the impact toughness stabilizes. These results taken with the microstructure observations show that 740H does have sufficient stability to serve in power plant applications.



700°C (1292°F), 750°C (1382°F) and 800°C (1472°F); a) Tube - 50 mm (1.96 in.) x 8 mm (0.31 in) and b) Pipe - 358 mm (14.1 in) x 88 mm (3.46 in.). Tube test specimens half-size .

High-Temperature Corrosion Properties

Materials used for high temperature process tube and pipe must be able to withstand a variety of corrosive environments if they are to be used uncoated. Alloy 740H was initially designed for use in boiler tubes which must have superior resistance to hot corrosion in the presence of coal ash deposits on the OD and oxidation by steam on the ID. Subsequently, a considerable amount of characterization work has been carried out in Europe and USA for behavior under these conditions. Evaluation in other corrosive environments has been less thorough, but by virtue of its 25% Cr content, alloy 740H should have substantial resistance to many oxidizing environments.

Oxidation in Air and Steam

Alloy 740H is resistant to scaling in the presence of high-temperature dry air and steam. This has been confirmed by independent studies in the USA and Europe. This work that was summarized by Smith and Baker [Smith 2014], showed parabolic weight change in the range 650°C (1202°F) – 800°C (1472°F) in testing up to 10,000 h with no observed oxide spallation. Optical and scanning electron metallography performed on long time oxidation test samples showed that matrix Cr is sufficient to replenish the scale and that internal attack is minimal.

Sarver and Tanzosh [Sarver 2010] have characterized candidate nickel-base alloys for A-USC service in a program sponsored by the U.S. AUSC Consortium. They developed rate constant data for various candidate A-USC alloys, including 740H at 650°C (1202°F), 700°C (1292°F) and 800°C (1472°F). The steam oxidation testing was performed using high purity water with 100-300 ppb of dissolved oxygen and 20-70 ppb of ammonia to produce a pH value of 8.0-8.5 and a pressure of 17 bar. These authors reported the chromium level at the alloy-oxide interface for alloy 740H as 17.4% after 4,000 h at 800°C (1472°F). Depletion extended inward to a depth of 56 µm. Al and Si enrichment was found just below the Cr₂O₃ scale. Internal grain boundary penetration after 4,000 h appears negligible at 650°C (1202°F), approximately 20 µm at 700°C (1292°F) and greater than 32 µm at 800°C (1472°F). In this test alloy 740H had slightly lower mass gain than alloy 617.

Coal Ash Corrosion

While resistance to steam oxidation is largely a matter of having sufficient Cr, resistance to coal ash corrosion is more complicated. Refractory metals such as Mo and W promote breakdown of protective chromia films in contact with sulfate and chloride-rich ash deposits. Alloy 617 with 9% Mo has very high rate of attack compared with alloy 740 that has only 0.5% Mo, while alloy 263 with 5.8% Mo falls between the two. Independent studies of alloy 740H corrosion in a variety of coal ash conditions have been conducted [10]. A new more comprehensive study that uses residue from actual ash deposits from ten commercial grades of coal is sponsored by the US Department of Energy. At this point it appears that alloy 740 has sufficient resistance to serve uncoated as a boiler tube, but validation under ash deposit conditions of the expected operating environment is advised.

A recent experiment compared welded and unwelded samples in a simulated Chinese coal ash deposit [Baker 2013]. Testing was performed at 750°C (1382°F), exposing coupons having a painted-on slurry comprised of a simulated ash containing 6% Fe₂O₃-29% CaSO₄-1.9% Na₂SO₄-1.9% K₂SO₄-39% SiO₂-22% Al₂O₃-0.05% KCl-0.05% NaCl. Components were pulverized and applied to sample surfaces as a slurry with acetone. Average mass gain via application of the slurry was 73 mg/cm². Sample dimensions were approximately 19 mm (0.75 in) x 12.7 mm (0.5 in) x 6.4 mm (0.25 in). A simulated flue gas comprised of N₂ – 15% CO₂ – 3.5% O₂ – 5% H₂O – 0.1% SO₂ was flowed over the samples at 500 sccm with platinum catalyst preceding the samples. Samples of both base metal and weld metal were tested (from GTAW welds made in matching base plates; alloy 740H samples were fabricated using matching welding wire. Cross sections of tested samples were made and evaluated, yielding corrosion depth results shown in Figure 5 for alloys 740H, 263 and 617. For alloys 740H and 263, both base metal and weld samples were tested after post-weld aging at 800°C (1472°F); the alloy 617 samples were tested in the solution annealed condition (2150°F/1177°C). Samples were exposed for 1000 h, with the ash coating re-applied at weekly intervals. Corrosion depths and corrosion product morphologies of base metal and weld metal were similar. External oxide scales were chromium-rich with low levels of titanium present in all cases. Alloy 740H formed a thinner, more compact chromia scale. All alloys developed internal oxides along

grain boundaries rich in Al/Ti. Chromium sulfides (verified with SEM-EDS and backscatter visualization), evident in the micrographs as medium-gray globular internal precipitates tending to reside on grain boundaries, were much more pronounced in the alloy 617 and 263 samples than in the alloy 740H samples. While the alloy 617 sample exhibited very significant pitting, the alloy 263 samples exhibited incipient shallow pit development. While alloy 740H had no pits, Natesan has proposed that nickel alloys exhibit an incubation period in this environment, so eventual pitting of 740H is possible.

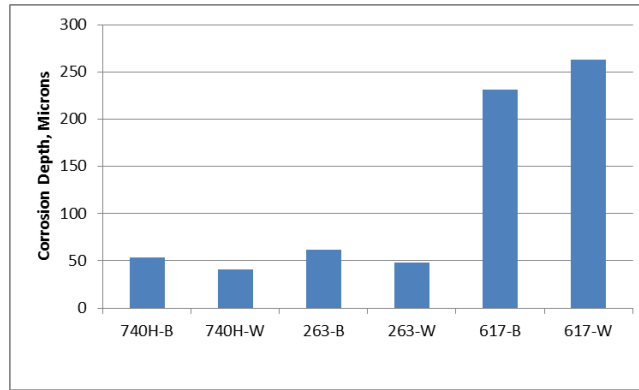


Figure 5. Corrosion pit depth for specimens exposed in a simulated Chinese coal ash. W designates welded samples.

Corrosion in Supercritical Carbon Dioxide

The Brayton cycle system, employing supercritical carbon dioxide as the working fluid, is under consideration for use in various power generation systems, including nuclear, solar, and fossil/biofuel. This system provides a means of achieving very high levels of efficiency at moderate temperatures. The University of Wisconsin-Madison has devised an apparatus to evaluate the corrosion behavior of metal samples in high high-pressure carbon dioxide at temperatures up to 650°C (1202°F) [Sridharan 2013]. Only a very limited set of test data has been published to date. The results indicate that 740H has a very low rate of mass gain; however, no information about performance at higher temperatures is available at this time.

Stress-Corrosion Cracking

Stress-corrosion cracking (SCC) would not be expected to occur under boiler operating conditions; however, during maintenance shutdown, the system may be exposed to condensed impure aqueous fluids. SCC has been observed in stainless steels in operating boilers. Experiments were conducted in a range of materials in standard accelerated tests to see if SCC might occur [Crum 2014]. The data reported here are extracted from a much larger list of materials that were tested. All materials were processed to 0.050" to 0.125" (1.32 mm to 3.18 mm) strip for testing. The age-hardened alloys were given standard anneals plus age-hardening heat treatments. The solid solution strengthened alloys were tested after annealing at standard solution annealing temperatures.

A summary of the boiling 45% magnesium chloride tests is given in Table IV. Alloys 617 and 347 stainless steel showed transgranular cracking while alloy 740H exhibited no cracking. Typically resistance to chloride SCC is strongly enhanced by increasing nickel content. The nickel content of all alloys tested was 50% to 59% except for and 347 SS, with 11% nickel. As expected the low-nickel 347 SS cracked severely in a transgranular mode.

Table IV. U-bend SCC test in boiling 45% Magnesium Chloride. 720 hr test duration. NC – no cracking, NF – no failure.

Alloy	Condition	Time to Crack, h	Time to Fail, h	Max Crack Depth, mm (mils)	Crack Morphology	Comments
740H	Aged	<720	NF 720	0	None	
		NC 720	NF 720	0	None	
617	Annealed	720	NF 720	0.05 (2)	Trans	
		NC 720	NF 720	0.02 (1)	Trans	
347	Annealed	5	24	2.97 (117)	Trans	Crack Full Thickness
		5	24	2.97 (117)	Trans	Crack Full Thickness

The test results obtained in boiling 50% sodium hydroxide are shown in Table V. All alloys developed very slight to severe transgranular cracking. The 347 was severely attacked by general corrosion, leaving a thick layer of corrosion product. Alloys 740H developed a light surface attack which could be regarded as a combination of rough general corrosion and SCC. The conclusion from these tests is that high-nickel age-hardened alloys including 740H are unlikely to experience SCC in power plant use.

Table V. U-bend SCC tests in boiling 50% sodium hydroxide. 720 h test duration. NC – no cracking, NF – no failure.

Alloy	Condition	Time to Crack, h	Time to Fail, h	Max Crack Depth, mm (mils)	Crack Morphology	Comments
740H	Aged	NC 720	NF 720	0	Trans	
	Aged	720	NF 720	0.02 (1)	Trans	
617	Annealed	720	NF 720	0.25 (10)	Trans	
	Annealed	720	NF 720	0.25 (10)	Trans	
347	Annealed	Stop 168 h			None	General Corrosion
	Annealed	Stop 168 h			None	General Corrosion

Manufacturing Demonstration

In the early stages of the AUSC program, superalloys were considered necessary only for superheater tubing that could be manufactured from small ingots made by the VIM/ESR melting process used for conventional aerospace applications. These ingots typically weigh 5000 kg (11,023 lb) or less. The first 50 mm (1.97 in) OD x 10 mm (0.39 in) W tubing was made under the Thermie program. This tubing was made by extruding billet, cut from forged bar, to tube shell and cold working to size using multiple cold work and anneal cycles. This tube process is similar to that used for other “hard” alloys such as 625 and 718. However, in the USA AUSC project with its planned higher operating temperature, superalloys would be needed for the much larger steam transfer pipes and associated connections. It has been determined that solid solution alloys such as alloy 617 are less economical because of the excessively heavy wall required for these lower strength materials [Shingledecker 2012], and that age-hardened alloys would be required. These structures are extremely large; some steel reheater pipes in current boiler designs are 1000 mm (39 in) OD with 100 mm (3.9 in) wall. These pipes will require ingots on a size scale never previously made with γ strengthened alloys. The first demonstration heavy-wall superalloy pipe was extruded from alloy 263 in 2002 at Wyman-Gordon in Livingston, Scotland. Although this relatively short pipe was made from a conventional size ingot, it demonstrated manufacturing feasibility of making heavy wall pipe. Subsequently a larger pipe of alloy 740H was extruded at Wyman-Gordon Houston TX [Klingensmith 2011]. This 378 mm (14.9 in) OD x 88 mm (3.5 in) W x 10.5 m (34 ft) L pipe is shown in Fig 6. A 762 mm (30 in) diameter, 7570 kg (6,688 lb) VIM/VAR Ingot was used for this extrusion. VAR was selected as the remelt step in order to minimize the risk of solidification segregation. Careful thermal profile management was necessary to prevent ingot thermal stress cracking. Excellent chemistry uniformity and microstructure was obtained. The detail for this work has been reported previously [deBarbadillo 2014]. Based on this trial it was projected that a 740H pipe as large as 750 mm (29.5 in) diameter could be extruded on the Wyman-Gordon press.



Figure 6. Heavy wall 740H pipe extruded at Wyman-Gordon, Houston TX.

Tube Bending:

Coal fired boilers are designed with a range of small and large radius tube bends. Normally bending is done in the shop of the designer/fabricator. Tube bending can be done cold or hot over a mandrel or by hot extrusion. Several fabricators have successfully made bends in alloy 740H tubing however very little of this information has been published. Most of the trials have involved cold bending of material that is in the solution annealed condition. In that condition, the alloy is relatively soft and ductile. Tensile properties of solution annealed tubing produced at two different Special Metals locations are shown in Table I. The yield and tensile strength and ductility are similar to that of solution annealed alloys 617 and 625. Alloy 740H does exhibit strong work hardening, but slightly less than alloy 625.

While cold bending of solution annealed 740H has proven rather trouble-free, cold bending of aged material is much more difficult due to greater spring back, inconsistent wall dimensional control and potential for strain-age cracking. The ASME code case requires the tube manufacturer to certify the material properties in the annealed and aged condition. This poses a dilemma for the fabricator because a re-anneal would be required before bending. The possibility of certifying solution annealed tube with a capability aging treatment and mechanical test is being explored.

Code case 2702 also requires a full solution anneal before age hardening if the cold work exceeds 5%. Several independent studies have shown that alloy 740H shows a loss of creep strength when cold strained in the range of 5-15%. Shingledecker, working with the U.S. A-USC Consortium has conducted a detailed investigation of the effect of cold work during bending on creep-rupture properties of directly aged alloy 740H and compared the results with literature data for other age hardened nickel-base alloys [Shingledecker 2013]. He found that the loss of creep strength is proportional to the amount of cold strain and that the mechanism of failure is enhanced void formation in the extrados region of the bend. At approximately 15% cold strain the creep strength falls outside of the scatter band for conventionally heat treated material. While there are conflicting reports for this effect in other age-hardened boiler tube alloys, it is quite likely that when compared under similar conditions, all materials will show creep strength degradation due to residual cold work. It should be emphasized that full solution anneal and age will restore the original creep strength of cold worked materials.

Pipe Bending

Large diameter pipes such as those for the header and re-heater in the boiler cannot be bent cold due to the excessive forces required. A commonly used manufacturing technique is hot induction bending. This process involves passing the pipe incrementally through an induction coil while simultaneously applying a bending moment. The method has been used to bend 740H boiler tubes; however, process detail, microstructure and properties have not been reported. To date hot bending of pipe has not been attempted. To demonstrate this capability, Special Metals is collaborating with separately with two fabricators to perform controlled bends in a 324 mm (12.8 in) OD x 23 mm (0.88 in) wall pipe. Microstructure and mechanical properties will be evaluated and reported at a later date.

Fabricated Components

Small Forged Components

A number of small fittings such as flanges, nozzles, saddles, weldolets and some “tees” and “wyes” are required in power plant construction. If only a few parts are needed they can be machined directly from forged bar, but for large runs, they would be more economically made by press or hammer forging. In order to explore the response of alloy 740H, Special Metals is collaborating with fittings manufacturers in the US to produce and characterize some trial parts.

Figure 7a shows a prototype weld-neck flange produced at Maass Flange Corporation, Houston, TX, by hammer forging a 102 kg (225 lb), 375 mm (14.8 in) long mult cut from 200 mm (7.9 in) diameter forged bar. Two parts were made by upsetting, first on a hydraulic press, then hammer forging in three sessions, and finally punching out of the center. The initial temperature was 1176°C (2150°F) with finishing temperature of 1190°C (2175°F) to fill the corners. Excellent crack-free surface and corner fill was obtained.



Figure 7. a) Appearance of weld-neck flange forging; b) Etched cross-section

The forging was annealed at 1121°C (2050°F), water quenched and aged at 800°C (1472°F) prior to sectioning for metallographic examination. A macro-etched (etchant 70% HCl – 30% H₂O₂) section of one segment is shown in Figure 7b. The grain size of ASTM -1 to 1 is coarser than that obtained in tube and pipe. A somewhat lower forging temperature would be required to produce an average grain size of 2-3 that would provide the best balance of creep strength and impact toughness. Tensile properties shown in Table VI exceed ASME Code minimums. Two locations were tested, the horizontal base and the vertical web. Charpy V-notch impact toughness at these locations was very high. Two very short time creep-rupture tests have been conducted at 700°C (1292°F) at a stress of 482 MPa (70 ksi). The rupture lives of 71.8 and 80.5 h were relatively short but within the conventional 20% stress deviation range.

Table VI. Mechanical properties of heat-treated weld-neck flange forging.

Source	Sample	0.2% YS, MPa (ksi)	UTS, MPa (ksi)	El, %	RA, %	CVN, J (ft-lb)
ASME 2702	min.	620 (90)	1035 (150)	20		
Web	A	682 (99)	1043 (151)	44.8	36.1	147 (108)
	B	682 (99)	1048 (152)	43.9	32.3	138 (102)
Base	A	699 (101)	1064 (154)	48.6	42.2	172 (127)
	B	716 (104)	1064 (154)	44.3	41.5	194 (143)

The flange forging is representative of other simple shapes such as saddles and weldolets. Other parts such as “tees” and elbows involve more complex metal flow with significant stretching and greater die chill. Forgings of this type are planned.

Large Forged Components

Large pipe fittings and valve components must be press forged and will require extensive machining. Whereas small parts such as the flange previously described can be quickly heated and cooled and given optimum process conditions should exhibit microstructure and properties similar to pipe and tube, large forgings will exhibit a more complex structure. While no large forged part has yet been made from 740H, experience in producing large diameter pipe provides useful insight.

It was reported by Klingensmith that extrusion loads for nickel-base alloys limit the diameter and wall thickness of pipe that can be extruded. It is known that alloys with 8 to 15% refractory element content have especially high flow stress and as a consequence are more limited in extruded pipe size. Compressive flow stress data generated with solution treated bar specimens is shown in Figures 8a and 8b. The data show that 740H, although a stronger alloy when in the aged condition, has between 15% and 25% lower peak flow stress than alloy 617 over a range of strain rates and temperatures. This lower flow stress is important for manufacturing large forged shapes as well as large extruded pipes.

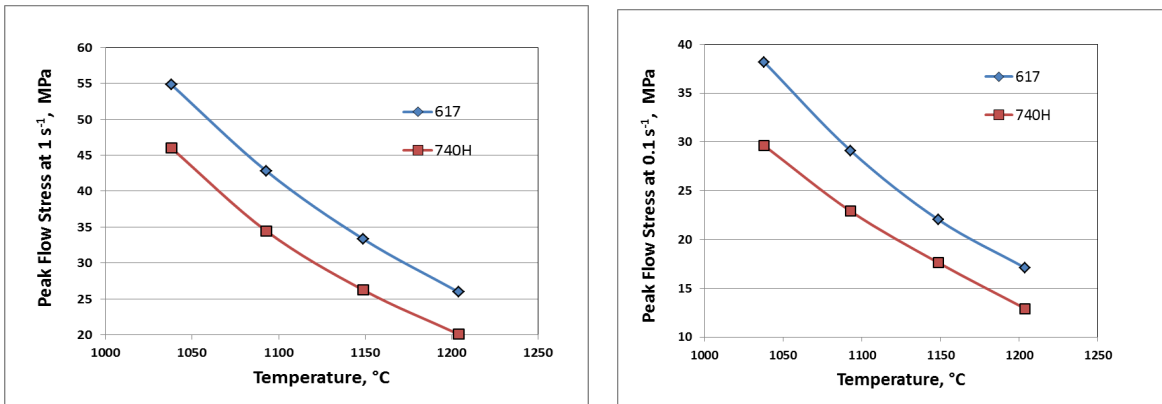


Figure 8. Peak compressive flow stress at strain rate of 1s⁻¹ (a) and 0.1s⁻¹ (b).

Very rapid cooling is not possible for massive “wyes”, “tees” and valve bodies that may be machined from block forgings. A calculated continuous cooling transformation diagram for alloy 740H is shown in Figure 9. This diagram indicates that significant γ hardening will occur even during water quenching of a large forging. For example, an air-cooled 450 mm (17.7 in) diameter extrusion billet had a hardness of Rc 36 compared with a solution treated tube hardness of Rb 90. For large parts that must be machined, it is recommended that a spheroidizing treatment of 24 h at 900°C (1652°F) be applied. This can be done either on cooling from forging, or as a re-heat treatment. Auto-aging will also affect the final heat-treated structure. Some coarse γ will form during cooling. This will produce a bi-modal γ structure after final aging. While this structure is produced deliberately in nickel-base superalloys for aircraft applications, it is usually generated through a two-step isothermal aging treatment. An experiment is now underway using a 325 mm (12.8 in) diameter forged bar to simulate heavy section microstructure and properties.

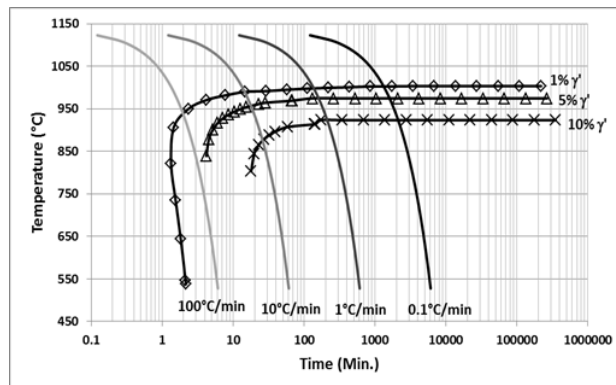


Figure 9. Calculated continuous cooling diagram for nominal composition of 740H (JMatPro software)

Welding

Fusion welding is an essential joining method for power plant construction. The initial welding studies on early versions of alloy 740H showed that it could be readily welded by GTAW and GMAW processes with good operability, tensile and bend properties. Successful girth welds were made on boiler tubes; however, extensive micro-fissuring was experienced in restrained heavy-section pipe welds. The redefined alloy 740H that has a reduced liquidus–solidus temperature range was shown to have excellent resistance to liquation cracking while retaining the operability characteristics of the original alloy. An SMAW electrode is not currently available for alloy 740H due to the difficulty of transferring Al and Ti through the flux. Experimental fluxes do exist for some other superalloys and it is expected that SMAW capability will ultimately be developed.

Process parameters were optimized by making butt welds on restrained 75 mm (3 in) thick plates. These parameters were then used to make circumferential welds on 378 mm (14.9) OD x 88 mm (3.46 in) wall extruded, solution annealed and aged pipe. These welds were made using a hot-wire narrow-groove GTAW process with a fixed torch and rotating work piece. Argon-25% He was used as the shielding gas for the matching composition filler wire. Welds were made with 5, 2, and 1 degree beveled V-grooves with a 1.57 mm (0.062 in) land. A cross-section of a weld with a 1 degree bevel is shown in Figure 10a. Note that the entire weld of 33 passes is one bead wide. The full pipe section was aged per ASME code requirement for 5 h at 800°C (1472°F) using a ceramic tile heating blanket. This method is commonly used for post-weld heat-treatment of field welds. No fissures, porosity or cracking were detected with ultra-sonic, radiographic or microscopic examination. Details of microstructure and weld qualification properties have been previously reported [Siefert 2010, Baker 2012]. The practical welding experience has been published [Gollihue 2013] and communicated in industry workshops. Shop-floor experience has been mixed due to unfamiliarity of welding this class of material. Key requirements for successful heavy section 740H welds are 1) use of proper shielding gas, 2) careful management of heat input and inter-pass temperature, 3) maintenance of correct bead geometry, and 4) frequent removal of residual surface oxides.

Once it was determined that sound welds could be made in heavy-wall pipe, a full scale header section was fabricated. For this simulated header, the nipples were inserted in predrilled holes and GTAW welded on the inside with a special rotary torch. The external welds were made manually by GTAW. This prototype header section is shown in Figure 10b.

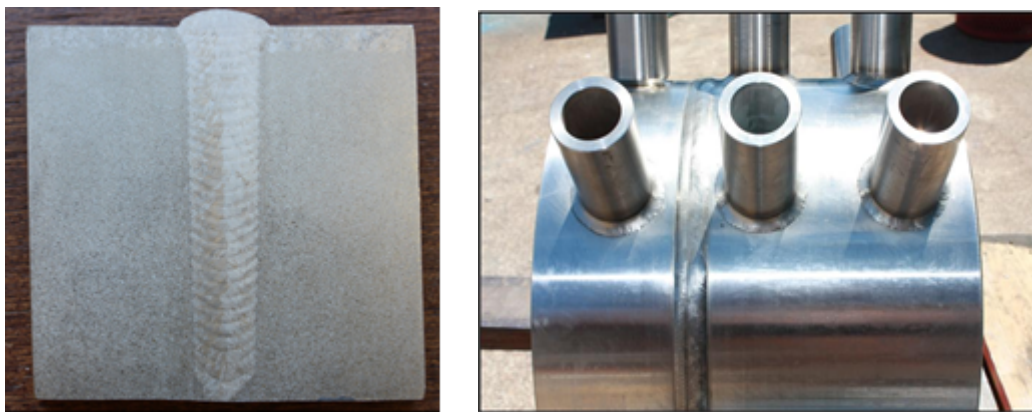


Figure 10. a) Macro graph of full-section pipe weld, b) Simulated header pipe.

Stress relief cracking is a well-known problem in γ' strengthened nickel-base alloys such as Waspaloy. Consequently, there has been considerable interest in how alloys such as 740H with a lower volume fraction of γ' will perform. Ramirez has investigated this issue using Gleeble tests and concluded that 740H is relatively resistant to this form of cracking (similar to alloy 718) [Siefert 2010]. To date, dozens of alloy 740H welds in 25 mm (1 in) to 75 mm (3 in) thick section have been made and stress relieved with no observed cracking. However, these short time tests cannot directly predict long-time behaviour of restrained structures in service. Special Metals is studying the problem using a Borland circular patch test

specimen exposed at 700°C (1292°F) [Borland 1960]. This test has been running for more than 5000 h. It has been examined with ultra-sonic and radiographic methods at 1000 h intervals. No indications have been observed (reference 0.38 mm for UST and 0.76 mm for radiography) to date. The test will be discontinued after 10,000 h and the weld sectioned and examined metallographically. Additional investigations of stress relaxation cracking in 740H and other nickel-base alloys are under way at Ohio State and Lehigh Universities, TNO, Utrecht Netherlands and by several fabricators.

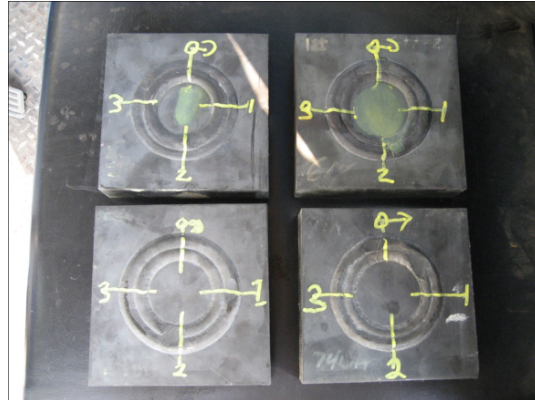


Figure 11. Appearance of 617 top) and 740H (bottom) Borland test specimens after 5000 h exposure at 700°C (1292°F)

Due to their high cost relative to steel, nickel alloys will only be used in sections of the A-USC boiler and turbine where they are absolutely essential. Consequently, they will need to be joined to other materials such as ferritic and austenitic stainless steels. Work by several fabricators is underway to evaluate dissimilar metal welds. A recent review of the results of welds to 316 and P92 steels was presented by Baker 2014]. Restrained, V-groove plate welds were made using GTAW (FM 82, P87 and 617) and SMAW (WE 182 and P87). The 740H was in the aged condition and welds were stress relieved for 4 h at 760°C (1400°F). All combinations produced sound welds and all except SMAW with WE P87 passed ASME tensile and bend qualification requirements with failure in the steel base metal. Creep tests are underway and results will be reported in the future. All creep specimens tested to date have failed in the steel member.

DISCUSSION AND CONCLUSIONS

An enormous amount of work to characterize the structure and properties of alloy 740H has been done in laboratories and plants in USA, Europe and Asia. This paper provides an overview of published information. But there is also a great deal of unpublished information, largely generated by power plant designers and fabricators some of which the authors are aware. Taken as a whole, this information confirms that alloy 740H has material properties and fabricability suitable for use for welded pressure containing components for power plants operating in the 650°C (1202°F) -850°C (1562°F) temperature range in a variety of corrosive environments. However, actual operating experience is very sparse. A few test loops containing 740H components have been constructed and several more are in the material procurement or fabrication stages. One full-scale plant application is in final installation stage. Clearly the feedback from installation, service and repair experience will provide important information for improving the technology of 740H as well as other age-hardened alloys that may be used in the future.

Several areas of technology need further development. Probably most important is a full understanding of stress relaxation cracking in complex restrained structures. Since this kind of cracking often takes thousands of hours to occur, a full characterization of the alloy will take some time. Experience under plant conditions is the real test. Other welding issues include 1) increasing weld creep strength reduction factor, 2) Developing a coated electrode for SMAW, 3) More thorough creep characterization of dissimilar metal welds and 4) Characterization of welds made in solution annealed base metal. Additional fabrication issues include characterization of hot induction bends and complex forgings that involved stretch metal flow.

While metal properties have been most extensively studied, some questions remain. These include mechanical and thermal fatigue and creep-fatigue properties and a more detailed understanding of damage tolerance. It would also be advisable to conduct hot corrosion tests in each specific environment for which the alloy might be considered.

NOMENCLATURE

A-USC = Advanced-Ultrasupercritical

EPRI = Electric Power Research Institute

ASM = American Society for Metals

ASME = American Society of Mechanical Engineers

ASTM = American Society for Testing and Materials

GTAW = Gas Tungsten Arc Welding

GMAW = Gas Metal Arc Welding

SMAW = Shielded Metal Arc Welding

UNS = Unified Numbering System

VIM = Vacuum Induction Melting

VAR = Vacuum Arc Remelting

ESR = Electro-slag Remelting

ID = Inside diameter

OD = Outside diameter

REFERENCES

ASME, "Case 2702 Seamless Ni-25Cr-20Co Material, Section 1, Cases of the ASME Boiler and Pressure Vessel Code, BPV-Supp. 7 2011, American Society of Mechanical Engineers.

Baker, B.A. and Smith, G.D., "Corrosion Resistance of Alloy 740 as Superheater Tubing in Coal-Fired Ultra-Supercritical Boilers", NACE Corrosion Conference, 2004, Paper No. 04526.

Baker, B.A., Smith, G.D., and Hazeldine, P., "Nickel-Base Material Options for Fabrication of Advanced Coal-Fired Boiler Components", NACE Corrosion Conference, 2009, Paper No. 09268.

Baker, B.A., Gollihue, R.D., de Barbadillo, J.J., "Fabrication and Heat Treatment of Weld Joints in Inconel Alloy 740H Superalloy Steam Header Pipe and Superheater Tubing" Welding and Repair Technology for Power Plants, Tenth International EPRI Conference, Marco Island, FL, June 26-29, 2012

Baker, B.A., Gollihue, R.D., de Barbadillo, J.J., Patel, S.J., and Maitra, D., "Manufacturing Demonstration of INCONEL Alloy 740H for AUSC Boilers", Proceedings: 7th International Conference on Advances in Materials Technology for Fossil Power Plants, EPRI, Waikaloa, HA, 2013, 215-229.

Borland, J.C., "Cracking Tests for Assessing Weldability", British Welding Journal, Vol. 7, 1960, 508-512.

Crum, J.R., and Maitra, D., "Stress Corrosion Cracking Resistance of Candidate AUSC Alloys", NACE Corrosion Conference 2014, Paper No. 3981.

de Barbadillo, J.J., Baker, B.A., and Xie, X., "Microstructure Stability of Alloy 740H and its Effect on Material Properties", Proceedings: ASME Symposium on Elevated Temperature Application of Materials for Fossil, Nuclear and Petrochemical Industries, ASME, Seattle, WA, March 25-27, 2014.

de Barbadillo, J.J., Baker, B.A., and Gollihue, R.D., "Age-Hardened Nickel-base Alloys for Advanced Energy Applications, 8th International Symposium on Superalloy 718 and Derivatives, Pittsburgh PA, Sept. 28-Oct. 1, 2014, to be published.

Gagliani, M.S., Hack, H., and Stanko, G., "Fireside Corrosion Resistance of Proposed USC Superheater and Reheater Materials"; Proceedings: 34th International Technical Conference on Clean Coal and Fuel Systems, Coal Technology Association, Clearwater, FL, May 2009.

Gollihue, R.D., Baker, B.A., Dierksheide, J.E., and Tanzosh, J.M., "Practical Guide to Welding INCONEL alloy 740H", Proceedings: 7th International Conference on Advances in Materials Technology for Fossil Power Plants, EPRI, Waikaloa, HA, 2013, 1025-1038.

Klingensmith, L., "Development of Extruded Heavy-Wall Large Diameter Nickel-Base Alloy Piping for AUSC Power Plants", Global Advanced Fossil Gen Summit 2011, Asian Coalition for Climate and Energy, Shanghai, China, Dec. 6-7, 2011, 272-298.

Sarver, J.M., and Tanzosh J., "Characterization of Steam-Formed Oxides on Candidate Materials for USC Boilers", Presented at Advances in Materials Technology for Fossil Power Plants - Proceedings from the Six th International Conference, August 31 - September 3, 2010, Santa Fe, NM.

Shingledecker, J.P., Hendrix, H., Siefert, J., Phillips, J., Purgert, R., and Rawls, P., "U.S. Program on Advanced Supercritical Power Plant Materials – The Economy of Using Advanced Alloys", IEA Clean Coal Centre Workshop: Advanced Ultrasupercritical Coal-fired Power Plants, Vienna, Austria, Sept. 19-20, 2012

Shingledecker, J.P., and Pharr, G.M., "Testing and Analysis of Full-Scale Creep-Rupture Experiments on INCONEL Alloy 740 Cold-Formed Tubing", Journal of Materials Engineering and Performance, Vol. 22, Issue 2, 2013, 454-462.

Shingledecker, J.P., "Creep-rupture Performance of INCONEL Alloy 740 and Welds", Proceedings: 7th International Conference on Advances in Material Technology for Fossil Power Plants, EPRI, Waikaloa, HA 2013, 230-241.

Siefert, J.A., Tanzosh, J.M., and Ramirez, "Weldability of INCONEL alloy 740H", Proceedings: Sixth International Conference in Advances in Materials Technology for Fossil Power Plants, Santa Fe, NM, Aug. 31 – Sept 3, 2010, 1045-1066.

Smith, R.J., Baker, B.A., and Smith, G.D., "Nickel Alloys for the Next Generatio of Advanced Steam Boilers", VGB Materials and Quality Assurance Conference, Dortmund, Ger., March 2004.

Sridharan, K., "Corrosion in supercritical Carbon Dioxide: Materials, Environmental Purity, Surface Treatments and Flow Issues," U. S. DOE Report 10-872, December 10, 2013, p. 205.

Wright, I., Appendix 2, Task 3b: Steamside Oxidation, "Boiler Materials for UltraSupercritical Coal Power Plants," U.S.. DOE No: DE-FG26-01NT41175, Phase 1 Final Summary Report, October 1, 2006 - December 31, 2006.

Zhao, S., Lin, F., Fu, R., Chi, C., and Xie, X., "Microstructure Evolution and Precipitates Stability in INCONEL Alloy 740H During Creep", Proceedings: 7th International Conference on Materials Technology for Fossil Power Plants, EPRI, Waikaloa, HA, 2013, 265-275.

ACKNOWLEDGEMENTS

The authors wish to acknowledge the leadership of John Shingledecker of EPRI and Jim Tanzosh of Babcock & Wilcox and continuing financial support of the USA A-USC Consortium for 740H characterization. We also acknowledge Maass Flange Company for collaboration on forging demonstrations.