
Steam Oxidation Testing of Candidate Ultrasupercritical Boiler Materials

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Abstract

Over the past several years, the electric power industry has recognized the need to increase operating temperatures and pressures of fossil power plants to increase efficiency and reduce pollutants. As temperatures increase, the ferritic materials presently being used in fossil boilers will not possess adequate steamside oxidation corrosion resistance. Ultrasupercritical boilers are being developed to operate with steam temperatures up to 760°C (1400°F), so new ferritic and austenitic materials must be utilized that possess excellent steamside oxidation resistance while meeting the required mechanical properties.

To this end, The Babcock & Wilcox Company (B&W), as a consortium member of the Department of Energy (DOE) / Ohio Coal Development Office (OCDO) "Boiler Materials for Ultrasupercritical Coal Power Plants" program, will perform steamside oxidation testing of candidate materials for ultrasupercritical boilers. Testing will be performed on ferritic and austenitic materials at temperatures ranging from 650°C (1202°F) to 900°C (1652°F). Specimens will be removed from the test at various times up to 4,000 hours. The specimens will be evaluated for weight change and scale morphology. Details regarding the test facility, test conditions, test materials, etc. are discussed. Also, results from a literature review on the steamside oxidation kinetics and behavior of ferritic, stainless and nickel-based alloys are presented. The literature review provided qualitative information regarding the steam oxidation behavior of these materials at ultrasupercritical temperatures, and approximate temperature usage limits for different classes of materials.

Introduction

The United States is facing a growing demand to produce more electricity, and at the same time permitted emissions from the power plants has been reduced so as to meet air quality standards. Power

plants are a major source of CO₂, SO₂ and NO_x emissions. The need to reduce these gases provides an additional incentive to increase efficiency. The European collaborative research programs in science and technology (COST) have made a considerable contribution to the improved efficiency and reliability of power generation plants through major advances in materials technologies.¹ The steam temperatures of the most efficient fossil power plants are now in the 600°C (1112°F) range, which represents an increase of about 60°C (108°F) in 30 years. It is expected that steam temperatures will rise another 50 to 100°C (90 to 180°F) in the next 30 years.² Currently, the power plants are typically 35 to 40% efficient. By developing better materials that can withstand higher temperatures, the DOE hopes to boost efficiencies to 52 to 55%. These efficiency gains, alone, would cut the release of CO₂ and other emissions by nearly 30 percent.³

The development of an ultra-efficient power generation system having low emissions of CO₂, SO₂ and NO_x poses a considerable challenge for the materials community. Therefore, this new effort taken by the DOE/OCDO is designed to help United States boiler manufacturers keep in step with advances that are being made in fossil power generation in Europe and Japan. Europe and Japan are experimenting with building a new line of supercritical steam generators with steam temperatures that would reach 620°C (1148°F). This goal would be achieved differently in the United States because so many aging coal plants have to be revitalized, and because the United States utilities will continue to burn coal under strict environmental regulations. Retrofitting existing plants with ultrasupercritical steam cycles is most definitely an option. But increasing temperatures up to 650°C (1202°F) would enhance efficiency by only a marginal amount, so the push is to create a class of pipes and tubing that can be used at temperatures of 760°C (1400°F) and possibly 871°C (1600°F).

Interest in the development of power plants with increased efficiencies has led to the consideration of materials capable of

operating at steam temperatures and pressures significantly higher than those employed in current power plants. The use of ferritic alloys can significantly reduce the cost of future ultrasupercritical steam generators and therefore, are of strong interest. The cost of pressure parts represent about 20 percent⁴ of the total cost of the boiler, therefore the savings is significant. The key to meeting the requirements in supercritical conditions is to optimize the use of ferritic steels for pressure parts including thick-section components such as piping and headers. Numerous advanced ferritic alloys have been developed in the last few years. These alloys possess improved high temperature strength and thus higher temperature usage limits. As a result they can potentially be used to replace certain steam generator components typically constructed from austenitic steels for service at 620°C (1148°F). Ferritic steels can save on the costs of austenitic materials and also avoid problems associated with dissimilar metal welds (DMW) and differential thermal expansion. Consequently, considerable effort is being devoted worldwide to the development of ferritic steels with good strength and oxidation resistance at temperature up to 650°C (1202°F).

For higher service temperatures (above ~650°C [1202°F]), advanced austenitic steels or nickel-base "Super Alloy" will be needed. The alternative to austenitic steels is to use Ni-based alloys, although the associated increased cost must be justified. The main enabling technology is the development of stronger high-temperature materials, capable of operating under high stresses at ever increasing temperatures. The goal of the European COST-522 program on advanced steam power plants is to identify materials for use in steam at 650°C/296 atm. (1202°F/4350 psi) while the Japanese national program has a goal of 650°C/349 atm. (1202°F/5135 psi). A demonstration program on this issue was also launched in 1998 by a group of 40 European utilities, research laboratories and equipment manufacturers with economic support from the European Commission's THERMIE program. Commissioning of the advanced plant is foreseen for the year 2010 and the Thermie program has aimed at 700°C/388 atm (1292°F/5440 psi) steam and net efficiencies of 52-55%, depending on site and fuel conditions. Further developments of steels, coatings and surface treatments are in progress.¹

The major materials property requirements for the high temperature components are primarily creep, fireside corrosion, and thermal fatigue resistance. Equally important is steamside oxidation of tubes and exfoliation of the oxide scale. Exfoliation is expected to be more severe in advanced steam plants, because the much higher steam temperatures employed are likely to cause more rapid formation of oxide scale. As a consequence, solid-particle erosion damage to the turbine can occur.

Steamside Oxidation Literature Review

The objective of the steamside oxidation literature review was to evaluate the oxidation kinetics and the steam oxidation resistance of the current and new high temperature alloys that could potentially be used in the ultrasupercritical coal power plants where boiler could be operated up to steam conditions of 760°C (1400°F) and 38 MPa (about 5500 psig). Limited information was found for the alloys of interest at these temperatures and pressures.

Steamside oxidation of tubes and exfoliation of the oxide scale is expected to be more severe in advanced steam plants. As is known, ferritic alloys exhibit less oxidation resistance to steam than austenitic steels due to their relatively low chromium levels. Practical temperature limits exist for the ferritic alloys, above which the alloys have limited life. Fe-Cr alloys containing 2-3%Cr are limited

in boiler service to temperatures of 580 to 600°C (1076 - 1112°F), ferritic alloys in the 10 - 13%Cr class appear to be capable of use up to approximately 620°C (1148°F).⁵ The temperature limits on the 300-series stainless steels (17 - 19%Cr) range from 700°C (1292°F) up to the ASME Boiler and Pressure Vessel Code creep rupture-based limit of 816°C (1500°F).⁶ Likewise, Ni-alloys can also be used at temperatures in excess of 700°C (1292°F).

When the metal temperature is below approximately 580°C (1076°F) and a sufficiently high partial pressure of oxygen is present, a double-layer scale consisting of magnetite (Fe₃O₄) and hematite (Fe₂O₃) is found on the steamside surface of ferritic alloys. Most of the oxidation studies in steam suggest that parabolic kinetics prevail at temperatures up to 700°C (1292°F); however, as a result of the formation of multilayered scales, the growth rate changes to linear above this temperature.^{7,8} Earlier studies of model Fe-Cr alloys suggested that linear kinetics prevailed at temperatures above 700°C (1292°F) for Cr levels in the range of 1-15 percent.⁷ In ferritic steels with chromium concentrations between 2 and 9%, corrosion behavior at temperature less than 600°C (1112°F) was independent of chromium content, but at higher temperature, a clear dependence of oxidation rate on alloy composition was observed.^{9,10} The scaling resistance of the alloys in high temperature steam increased with the Cr content of the alloys. The corrosion rate of 12%Cr steel was at about half of that for 9%Cr steel at 650°C (1202°F).¹¹ Alloys containing more than 20% Cr and fine grains have better scaling resistance when tested at 532°C (990°F) for 1000 hrs than the conventional coarse-grained stainless steel.¹² Likewise, high-Cr, high-Ni alloys 807, 800H and HR3 have better scaling resistance than the stainless steels. No scale was formed on the HR3 alloy containing 24% Cr. Little to no scale was observed on nickel alloys Inconel 617, Inconel 625, Inconel X and SZ alloy when exposed to 700°C (1292°F) steam for 1000 hrs.¹³

The rate of oxidation can also be affected by the alloying elements. The element most widely used for improving oxidation resistance of steels is chromium. In ferritic steels the oxidation resistance can also be improved by addition of silicon and sulfur. The amount of about 0.3% Si, is beneficial to steamside oxidation above 500°C (932°F).¹⁴ The sulfur content should be in the range of 0.005 % to 0.01% in high Cr ferritic steels to improve the oxidation resistance without producing any harmful effect on the mechanical properties.¹⁵

Most of the experimental work on steam oxidation was done at steam pressures less than 0.1 MPa (14.5 psig), whereas, supercritical steam conditions would require pressure greater than 21 MPa (3075 psig). Only a small effect of pressure over the range 1 to 10 MPa (145 to 1450 psig) was observed on the oxidation rates of 2% and 9-11% Cr alloys at 700°C (1292°F), while at 600°C (1112°F) and below the value of the oxidation rate constant decreased with increasing pressure.¹⁶ In low pressure studies of Ar-water vapor mixtures, a strong pressure dependence in the temperature range 800 to 1100°C (1472 to 2012°F) was reported, with the oxidation rates increasing when the pressure was increased.⁷ Surface pretreatment also affects the corrosion resistance of the alloys. An increase in cold work increases the corrosion resistance due to improved diffusivity of chromium in the defect structure, preferential oxidation, and formation of a solid solution (FeFe_{2-x}Cr_xO₄) of spinel type oxide scale.¹⁷

A common finding in many of the studies of oxidation in steam or water vapor is that the scales exhibit significant porosity, to the extent that the suggested oxidation mechanisms invoke transport of Fe out and steam in via an interconnected pore network. The oxide

scale morphology varies with the alloy composition. In steels containing chromium, two types of oxide layers are formed; chromium oxide (Cr_2O_3) with excellent protective properties and a spinel (FeCr_3O_4 , which is less protective).¹⁸ The inner layer of an 11% Cr alloy exposed to 700°C/10 MPa (1292°F/1450 psig) steam contained over 40% Cr, which provided an effective diffusion barrier.¹⁶ In the lower chromium alloys, double-layered magnetite scales have been reported, where the ratio of the thicknesses of the inner and outer layers was independent of time, but increased with increasing alloy chromium content. Scales formed on austenitic Cr-Ni steels are similar to those formed on ferritic steels except that they are usually thinner and contain less alloying elements in the inner magnetite layer.¹⁹

The exfoliation of steamside scale which is common to both ferritic alloys and austenitic stainless steels is associated with the stress in the scale.²⁰ Exfoliation could become worse in the advanced steam plants because of increased oxidation rates due to the higher steam temperature. The differences in the coefficients of expansion between the various scales and the base metal have been identified by a number of researchers as being significant with respect to the magnitude of strain acting on the scale when it is cooled.²¹ Exfoliation occurs in some cases near the metal/scale interface and in other cases at the interface between the outer and inner part of the scale, depending on the differences in coalescence of pores present in the scales.¹⁰ A substantial reduction of scale growth and resistance to exfoliation was obtained by using aqueous chromate treatment.²⁰ The chromate pretreatment resulted in a scale containing an iron-chromium spinel at the mid-band of the scale. Higher chromium austenitic alloys exfoliate to a lesser extent and only the outer magnetite layer is lost.⁶ As a result, the overall oxidation rate of these alloys in steam is closer to parabolic than linear.

There are numerous worldwide research activities that are currently in progress to develop and implement a class of unique ferritic steels containing 9 - 12%Cr.² High-strength ferritic 9-12Cr steels for use in thick section components are now commercially available for temperatures up to 620°C (1148°F). For higher temperature service, austenitic steels and Ni-based alloys are needed. Advanced austenitic stainless steels (such as 319CbN, NF709 and SAVE25) for use as super and reheater tubing are available for service temperatures up to 650°C (1202°F) and possibly 700°C (1292°F). Ni-based superalloys would be needed for higher temperatures.

The results from this literature review indicate that uncoated ferritic materials cannot be used in the highest temperature regions of an ultrasupercritical power plant. Based on this result, the majority of ferritic materials that will be included in the B&W steam oxidation test will only be tested at 650°C (1202°F). New ferritic materials will be included in the B&W steam oxidation tests to determine if the maximum temperature usage limit for these materials exceeds that of other ferritic materials. Austenitic materials (stainless steels and nickel-based alloys) will be employed in the highest temperature regions of an ultrasupercritical power plant, and will be tested at temperatures up to 900°C (1652°F) in the B&W steam oxidation tests. The results from this review will be used to guide the selection of the austenitic materials that will be included in the steam oxidation test program.

Planned Test Program

A test program has been developed that will evaluate the steamside oxidation performance of candidate materials for ultrasupercritical service. This program will provide complimen-

tary results to the information available in the literature, and, in conjunction with the results from other Tasks in this program, will provide a firm basis for material selection choices. Details of the test program are described in the following sections.

Test Parameters

To bracket the anticipated maximum operating temperature of a USC plant (760°C [1400°F]), the steamside oxidation tests will be performed at temperatures of 650°C (1202°F), 800°C (1472°F) and 900°C (1652°F). Testing will be performed on coupons in slowly flowing steam at near atmospheric pressure. The total exposure time at each temperature will be 4,000 hours, with interim specimen removals after 1,000 and 2,000 hours.

Test Facility

A schematic diagram of the test facility is shown in Figure 1. Test solution from one of two feedtanks will be pumped by a peristaltic pump through Alloy 600 tubing. The tubing runs from the pump and into a furnace. The test solution will flash from liquid to steam in the tubing that is inside the furnace. The tubing inside the furnace is connected to a 310SS retort (~5.75" diameter x ~21" length) which houses the test specimens. By the time the steam enters the retort, it is at the desired test temperature. Alloy 600 tubing that is connected to the other end of the retort exits the furnace and is connected to a cooling coil which condenses the steam back to liquid. An on-line oxygen probe will continuously monitor the oxygen content of the condensed steam at this location. The condensed steam then flows to drain.

The use of two controlled environment stainless steel feedtanks allows one feedtank to supply test solution to the facility, while test solution in the second feedtank is being prepared and analyzed. This approach insures a continuous supply of a consistent test chemistry.

The 310SS retort has been pre-oxidized in high purity steam prior to the initial steamside oxidation test to reduce the oxygen consumption by the vessel. Inside the retort, an Alloy 601 frame will hold two ceramic test racks (one above the other) in which the coupons will be placed. The coupons will be oriented in the test racks parallel to the steam flow.

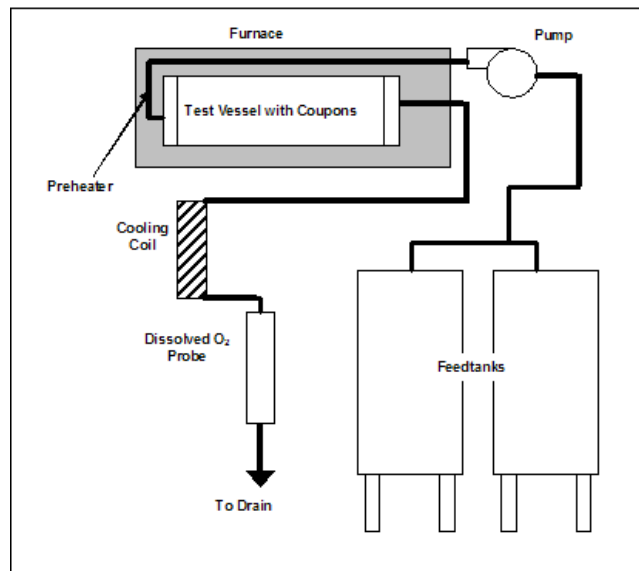


Fig. 1 Schematic of steamside oxidation test facility.

Test Environment

The test environment is high purity water containing 20-70 ppb ammonia to maintain a pH between 8.0 and 8.5. This environment is continuously recirculated within the feedtank. The feedtank contains a slight overpressure of an argon-0.64% oxygen cover gas to insure that the water being used in the test contains 100-150 ppb of dissolved oxygen. The water chemistry thus meets the specification for oxygenated treatment (OT) fossil boiler water.

During heat-up to, and cool-down from the desired test temperature, pure argon gas will flow through the retort to minimize oxidation of the specimens and eliminate condensation of steam within the retort. At the beginning of a test run, steam will be introduced to the retort once the temperature has stabilized within the retort. As of this writing, the exact flow rate of the test solution has not been decided. It is anticipated that a flow rate of approximately 20 ml/min will be used. Trials will be performed to determine how flow rate affects the oxygen content of the steam that exits the retort.

Test Materials

It appears that no currently available ferritic alloy possesses the steamside oxidation (or fireside corrosion) resistance to withstand the highest temperature regions of ultrasupercritical boilers. For these regions, austenitic alloys or ferritic alloys with specialized coatings must be employed. However, to maximize the economic benefit of generating electricity under ultrasupercritical conditions, ferritic materials must be used at their highest usable temperature. Thus, the steamside oxidation test program will evaluate both ferritic and austenitic materials. These tests will provide valuable information regarding the usable temperature limits and steam oxidation kinetics for several different materials.

The list of test materials has not yet been finalized, however, as of this writing; the planned list of materials is shown in Table 1. As can be seen in Table 1, the same ten "baseline" materials will be tested at each temperature. These baseline materials include advanced ferritic materials, iron-based austenitic alloys and nickel-based alloys. In addition to the baseline alloys, six coated materials will be included at each temperature. The remaining four test slots at each temperature will be filled with other materials of interest, with austenitic materials filling more of the test slots as the test temperature increases. The composition of all of the currently planned materials is shown in Table 2.

Some of the criteria used to select the materials in Table 2 included: favorable steamside oxidation results under conditions that approached ultrasupercritical conditions, previous experience with the material in fossil boiler applications, and the possession of other material properties (e.g. creep resistance) that are necessary for ultrasupercritical service.

Evaluation

Six coupons ($\sim\frac{1}{2}$ " x 1" x $\frac{1}{4}$ ") will be tested from each material at each temperature. The coupons will be measured and weighed prior to testing. At each shutdown (after 1,000, 2,000 and 4,000 hours), two coupons from each material will be removed. After removal, one of the coupons from each material will be cross sectioned and metallurgically examined to determine oxide morphology and composition. The other coupon from each material will be weighed, descaled, and re-weighed to determine the weight change (and, thus, oxidation rate) for each material.

Table 1
Planned Steamside Oxidation Test Materials

Slot	650°C (1202°F)	800°C (1472°F)	900°C (1652°F)
1	P92 (F)	P92 (F)	P92 (F)
2	230 (A)	230 (A)	230 (A)
3	HR-120 (A)	HR-120 (A)	HR-120 (A)
4	SAVE 25 (A)	SAVE 25 (A)	SAVE 25 (A)
5	Alloy 617 (A)	Alloy 617 (A)	Alloy 617 (A)
6	Super 304H (A)	Super 304H (A)	Super 304H (A)
7	Alloy 740 (A)	Alloy 740 (A)	Alloy 740 (A)
8	HR6W (A)	HR6W (A)	HR6W (A)
9	SAVE 12 (F)	SAVE 12 (F)	SAVE 12 (F)
10	304H (A)	304H (A)	304H (A)
11	T23 (F)	T23 (F)	Nimonic 263 (A)
12	"9Cr" (F)	214 (A)	214 (A)
13	Nimonic 263 (A)	310HCbN (A)	310HCbN (A)
14	800HT (A)	347HFG (A)	RA602CA (A)
15	Coated 1	Coated 1	Coated 1
16	Coated 2	Coated 2	Coated 2
17	Coated 3	Coated 3	Coated 3
18	Coated 4	Coated 4	Coated 4
19	Coated 5	Coated 5	Coated 5
20	Coated 6	Coated 6	Coated 6

Summary

The proposed operating conditions for an ultrasupercritical fossil boiler present many materials-related challenges. Through careful consideration of previous test results and operating experience, and from the results of planned steamside oxidation tests in the DOE/OCDO program, the steamside oxidation behavior of classes of materials, as well as individual alloys, will be characterized at ultrasupercritical temperatures. This information will be considered, along with the results from other portions of the DOE/OCDO program, to facilitate the process of material selection for all of the components within an ultrasupercritical boiler.

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Table 2
CHEMICAL COMPOSITION OF TEST MATERIALS

Material	C	Si	Mn	Fe	Cr	Ni	Mo	W	V	Nb	N	Other
T23	0.06	0.20	0.45	Bal	2.25		0.10	1.6	0.25	0.05		0.003B
P92	0.07	0.06	0.45	Bal	9.0	0.04	0.5	1.8	0.20	0.05	0.06	0.004B
9Cr	0.08	0.30		Bal	9.0			3.0	0.20	0.05	<0.003	
SAVE 12	0.10	0.3	0.20	Bal	11.0			3.0	0.20	0.07	0.04	0.04Nd, 3.0Co
304H	0.08	0.6	1.6	Bal	18.0	8.0						
Super 304H	0.10	0.27	0.78	Bal	18.15	9.25				0.40	0.091	3.0 Cu
347HFG	0.08	0.6	1.6	Bal	18.0	10.0				0.8		
310HCbN	0.06	0.4	1.2	Bal	25.0	20.0				0.45	0.2	
HR6W	0.07	0.16	1.12	Bal	23.15	41.87		5.47		0.18		0.08Ti
Alloy 800HT	0.08	0.5	1.2	Bal	21.0	32.0						0.5Ti, 0.4Al
SAVE 25	0.10	0.1	1.0	Bal	23.0	18.0		1.5		0.45	0.2	3.0Cu
214	0.05			3.0	16.0	75.0	0.50	0.50				0.01 max B, 4.5Al, 2Co, 0.01Y
Alloy 230	0.10	0.4	0.5	3.0 max	22.0	57.0	2.0	14.0				5.0 max Co, 0.3 Al, 0.02 La 0.015 max. B.
HR-120	0.05	0.60	0.70	33.0	25.0	37.0	2.5			0.70	0.20	0.1Al, 3.0Co
Alloy 740	0.06	0.50	0.30	0.7	25.0	48.0	0.5					0.9Al, 20Co, 1.8Ti
Alloy 617	0.07	0.40	0.4	1.5	22.0	54.0	9.0					12.5Co, 1.0Al, 0.3Ti,
RA602CA	0.02	0.5 max	0.15max	9.5	25.0	Bal.						2.1 Al, 9.5 Fe, 0.15Ti, 0.08Y, 0.05Zr
Nimonic 263	0.06	0.4 max	0.6 max	0.7 max	20.0	Bal.	5.9					20.0 Co, 2.2 Ti, 0.6 max Al, 0.005 max B, 0.2 max Cu

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