

# Quarterly Progress Report

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## **Abstract**

The U.S. Department of Energy (DOE) and the Ohio Coal Development Office (OCDO) have recently initiated a project aimed at identifying, evaluating, and qualifying the materials needed for the construction of the critical components of coal-fired boilers capable of operating at much higher efficiencies than current generation of supercritical plants. This increased efficiency is expected to be achieved principally through the use of ultrasupercritical steam conditions (USC). A limiting factor in this can be the materials of construction. The project goal is to assess/develop materials technology that will enable achieving turbine throttle steam conditions of 760°C (1400°F)/35 MPa (5000 psi). This goal seems achievable based on a preliminary assessment of material capabilities. The project is further intended to build further upon the alloy development and evaluation programs that have been carried out in Europe and Japan. Those programs have identified ferritic steels capable of meeting the strength requirements of USC plants up to approximately 620°C (1150°F) and nickel-based alloys suitable up to 700°C (1300°F). In this project, the maximum temperature capabilities of these and other available high-temperature alloys are being assessed to provide a basis for materials selection and application under a range of conditions prevailing in the boiler. A major effort involving 9 tasks was recently completed in phase 1.. In this phase 2, the earlier defined tasks have been extended to finish and enhance the phase 1 activities. In addition, preliminary work has been undertaken to model an Oxyfuel boiler to define local environments expected to occur and to study corrosion behavior of alloys under these conditions This report provides a quarterly status report for the period of July1 – September 30, 2007.

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# 1.0 Executive Summary

## A. Project Objective

The principal objective of this project is to develop materials technology for use in ultrasupercritical (USC) plant boilers capable of operating with 760°C (1400°F), 35 MPa (5000 psi) steam.

## B. Background and Relevance

In the 21st century, the world faces the critical challenge of providing abundant, cheap electricity to meet the needs of a growing global population while at the same time preserving environmental values. Most studies of this issue conclude that a robust portfolio of generation technologies and fuels should be developed to assure that the United States will have adequate electricity supplies in a variety of possible future scenarios.

The use of coal for electricity generation poses a unique set of challenges. On the one hand, coal is plentiful and available at low cost in much of the world, notably in the U.S., China, and India. Countries with large coal reserves will want to develop them to foster economic growth and energy security. On the other hand, traditional methods of coal combustion emit pollutants and CO<sub>2</sub> at high levels relative to other generation options. Maintaining coal as a generation option in the 21st century will require methods for addressing these environmental issues.

This project has established a government/industry consortium to undertake a five-year effort to evaluate and develop advanced materials that allow the use of advanced steam cycles in coal-based power plants. These advanced cycles, with steam temperatures up to 760°C, will increase the efficiency of coal-fired boilers from an average of 35% efficiency (current domestic fleet) to 47% (HHV). This efficiency increase will enable coal-fired power plants to generate electricity at competitive rates (irrespective of fuel costs) while reducing CO<sub>2</sub> and other fuel-related emissions by as much as 29%.

Success in achieving these objectives will support a number of broader goals. First, from a national prospective, the program will identify advanced materials that will make it possible to maintain a cost-competitive, environmentally acceptable coal-based electric generation option. High sulfur coals will specifically benefit in this respect by having these advanced materials evaluated in high-sulfur coal firing conditions and from the significant reductions in waste generation inherent in the increased operational efficiency. Second, from a national

prospective, the results of this program will enable domestic boiler manufacturers to successfully compete in world markets for building high-efficiency coal-fired power plants

The current phase also includes exploring and modeling a oxy fuel boiler. Theoretical modeling as well as pilot studies will be conducted to evaluate the local environments at various locations in the boiler and their effect on corrosion of the materials of construction. The need to reduce CO<sub>2</sub> in the atmosphere has made oxygen fired boilers with flue gas recycling an attractive alternative to conventional coal fired boilers. By using oxygen as the oxidant and by recycling the flue gas, the concentration of CO<sub>2</sub> in the flue gas increases to approximately 85% to 90% by volume dry. This high concentration of CO<sub>2</sub> in the gas makes it an ideal candidate for sequestration. If this technology, commonly referred to as the oxy-fuel technology can be developed in an economical way, it would greatly reduce the amount of CO<sub>2</sub> emitted into the atmosphere. It also has the advantage of avoiding introduction of the nitrogen associated with air in the combustion process thus reducing the NO<sub>x</sub> generated in the combustion zone to levels that may significantly reduce or eliminate the need for selective catalytic reduction (SCR) equipment. The technology can be applied both as retrofit to existing boilers as well as to new boilers. In the latter case, the lower volume of flue gases that need to be handled can result in units that are markedly smaller in both size and in volume.

The project is based on an R&D plan developed by the Electric Power Research Institute and an industry consortium that supplements the recommendations of several DOE workshops on the subject of advanced materials. In view of the variety of skills and expertise required for the successful completion of the proposed work, a consortium that includes EPRI and the major domestic boiler manufacturers (Alstom Power, Babcock and Wilcox (a division of McDermott Technologies Inc.), Foster Wheeler, Riley Power Inc., and Oak Ridge National Labs) has been developed.

### C. Project Tasks

The original task structure from phase 1 has been retained, while adding the new activities as sub tasks to the existing ones as shown below:

<b>Task 1: Conceptual Designs</b>		
1C	Revisit USC conceptual designs	Alstom
1F	Revisit USC conceptual designs	BW
1A	Oxy-combustion, USC design	Riley
		BW
1E	Oxy-comb, USC design	(OCDO)
1B	Oxy-comb, USC design	Alstom

<b>Task 2: Material Properties</b>		
2J	Further characterization of In740	BW
2I	New materials evaluation	BW
	(ORNL efforts for these tasks are separately funded)	

<b>Task 3: Steamside Oxidation</b>		
3H	Steamside oxide exfoliation	BW
3J	Long term steam oxidation testing	BW
3I	Steam oxidation testing of weld metals/zones	BW

<b>Task 4: Fireside Corrosion</b>		
4F	Oxy-comb gas chemistry and related. corrosion	Alstom
4E	Steam loop testing at host site	Alstom
		BW
		FW
		Riley
		BW
4L	Oxy-comb modeling re. corrosion	(OCDO)
4H/1D	Corrosion in oxy-combustion boilers	FW
		BW
4K	Oxy-comb pilot testing for corrosion	(OCDO)
4G	Coal ash corrosion / carburization	Alstom
		BW
4I	Laboratory oxy corrosion testing	(OCDO)
4J	Investigate corrosive effect of CO2	BW
4D	Cold-end oxy-comb corrosion studies	Riley
was 3K	Cold-end oxy-comb corrosion studies	BW (OCDO)

<b>Task 5: Welding</b>		
5H	Potential for DMW failures in USC boilers	BW
5G	In72 weld overlay cladding study	BW

<b>Task 6: Fabricability</b>		
6E	Effect of cold strain on recrystallization	FW
6D	Fabricable water wall panels	Alstom

<b>Task 8: Design Data &amp; Rules</b>		
8E	Dynamic boiler response, cycling capability.	Alstom/Riley Riley
8F	Design guide/assessment. for welded components.	Alstom/Riley Riley
8G	Component durability test facility	Alstom



# ORNL Task 2: Mechanical Properties

## Background

The objective of Task 2 is to produce the mechanical properties database needed to design a boiler to operate at the steam conditions within the scope of the project. This requires: producing long-term creep data, understanding the microstructural development which effects long-term behavior, assessing creep-fatigue interactions, evaluating welds and welded joints, in-depth studies of processing variables (ex: cold-work) which may effect long-term component performance, and performing structural feature tests to validate/develop improved and simplified material/component models. As more understanding is gained about complex interactions between the processing, properties, and performance of the candidate alloys, the Task performs evaluations of the materials technology required for USC steam conditions up to 760°C and determines, from the perspective of long-term material and component behavior, if a candidate alloy/fabrication/joining process will meet the requirements of the program. If more research and development is needed, this is brought to the attention of the consortium.

The ORNL Task 2 work scope is jointly determined by ORNL and the consortium, and given in ORNL's field work proposal (FWP) which is funded directly by DOE.

## Progress

### Long-term creep strength

Long-term creep testing continues on base metal samples of Super 304H, CCA617, and Inconel 740. The longest test duration for the program is 37,000 hours (~4.2 years). For the CCA617, three tests continue beyond 20,000 hours and a number of ruptures have been in excess of 30,000 hours. Current ASME B&PV code requires data with a minimum of 6,000 hours rupture life, but rules are currently being considered to increase this to 10,000 or 20,000 hours. For the Super 304H and the CCA617, the long-term data re-enforce earlier analysis of these materials using shorter-term data.

The majority of the remaining base-metal creep-rupture testing is ongoing for Inconel 740. The goal of the testing is to produce the creep-rupture data required for an ASME Code Case for the material. Currently, one heat of tubing and one heat of 3" thick plate are being tested. A number of longer-term tests beyond 10,000 hours for these materials remain. One specimen ruptured after ~15,000 hours at 800°C this quarter. This was a promising result because the rupture time was consistent with the rupture predictions for the alloy based on shorter-term data. This provides evidence that even at 800°C, where the gamma prime coarsens rapidly and a significant amount of eta phase begins to precipitate, the material does not show a dramatic strength drop-off due to microstructural instability. The current status of the base metal creep testing is shown in figure 1 including this recent rupture. Specimens from a third heat (required for a code case) of bar product were manufactured this quarter and have been heat-treated. This testing will begin next quarter.

## **Microstructural Development**

Two types of microstructure studies are being conducted.

The first is to assess the microstructure development in the alloys and how this affects long-term strength, ductility, etc. This work utilizes electron microscopy (SEM, TEM, etc.) and is being performed by the University of Cincinnati, under a subcontract to ORNL. A draft paper has been produced on the characterization of long-term aged 617 and has been submitted to a journal for consideration. Writing has begun on a second paper detailing microstructure development in Inconel 740. Due to the decreases in FY2007 funding, the remaining funds on this subcontract were exhausted this quarter.

The second set of microstructure studies being conducted is to evaluate rupture characteristics and microstructural changes by optical microscopy. In the past, this has produced a failure map for HR6W on completion of the creep testing program and cracking behavior under different creep-fatigue conditions for CCA617. Currently, these activities are focused on evaluating the tube-bend tests to determine changes after high-temperature testing of cold-worked material.

## **Creep-Fatigue**

Previous data was analyzed and put on the website for CCA617 and Inconel 740. The CCA617 was also provided to Task 8 to help evaluate the thermal shock test. No testing has been performed for ~2 years on this activity.

## **Welds and Welded Joints**

The largest portion of the current testing has been focused on evaluating the long-term cross-weld creep-rupture strength of tube butt welds of Inconel 740 and CCA617, thin-plate Haynes 230, and thick-plate CCA617 (SMAW and SAW process).

Longer-term test data to ~6,000 hours confirm shorter-term rupture data on the thick plate CCA617 which indicated the SMAW process had better strength than the SAW process. Full-size specimens of both welding processes continue in ORNL's 50:1 and 200:1 lever-arm creep machines. These tests have expected durations of ~20,000 hours.

A number of new tests have been started on Inconel 740 GTAW tube butt welds for comparison with the base metal data and previously tested GMAW plate welds. The initial results to ~2,000 hours have been analyzed against the base metal data and the weld strength factors (WSF) for each test has been calculated. These data are plotted in figure 2 against testing time and temperature. No clear trend for WSF is observed when plotted against test temperature, but the WSF does appear to decrease with increasing test time. The average WSF for Inconel 740 is ~0.75 which is similar to other nickel-based alloys and is much better than advanced ferritic steels which can have a WSF of 0.5 in the fine-grained heat affected zone. For the 740 weldments, all the ruptures were in the weld. Clearly, longer-term testing is needed to determine if the WSF continues to decrease with time. Based on these findings, a number of cross-weld creep-rupture tests with expected durations of 10,000 hours have been initiated with test times progressing beyond ~1,000 hours.

## **Processing Variables**

The test set-up to perform the pressurized creep-rupture testing of cold-bent tubes to determine the effects of cold-work on component strength is ready for the next set of tube bends. The plans for testing either CCA617, re-testing of HR6W and Haynes 230, or testing the process of cold-bending and annealing Inconel 740, will be discussed at the next steering committee meeting. A paper detailing the results of the pressurized-creep testing of cold-bent boiler tubes of Haynes 230 and HR6W was completed this quarter and will be presented at the EPRI Marco Island conference next quarter.

## **Model Validation Tests**

Thermal shock testing continued on a second CCA617 specimen. The water pump on the induction heater cooling system broke this quarter and a replacement pump was ordered and received. The pump will be installed next quarter and testing will resume.

## **Accomplishments**

- Completed a paper for the EPRI Advanced Materials Conference (Oct. 2008, Marco Island, FL), on the effect of cold-work and recrystallization in cold-bent USC boiler tubing.
- Creep-rupture tests for times up to 15,000 hours were completed on Inconel 740. These rupture results did not indicate any unexpected strength reduction at 800°C. Testing continues for times exceeding 17,000 hours and specimens from a third heat of material have been prepared.
- An initial analysis of the weldment strength of Inconel 740 was conducted. The average weld strength factor (WSF) was found to be 0.75 which is similar to other nickel-based alloys. However, the data suggest this factor may decrease with testing time, so longer-term tests have been initiated.

## **Concerns**

FY2007 funding was significantly reduced causing a reduction in ORNL's scope. The effects of this reduction and the plans for utilization of the current budget have been presented to consortium who has agreed with ORNL's FY2007 plan.

## **Plans for Next Quarter**

Continue all current testing including base metal and weldments. Initiate testing on a third heat of Inconel 740. Present a paper at the EPRI materials conference. Repair induction heating unit and resume thermal shock testing.

## **ORNL Task 3: Steamside Oxidation**

### **Progress**

No progress this quarter as funding has been exhausted.

### **Concerns**

The reduced level of FY2007 funding available for ORNL's Task 2 effort forced the decision to first complete the analysis of previously-exposed specimens. The remaining funding is insufficient to cover the cost of analysis of the recently-completed exposure at 800°C, or to prepare specimens and run the planned exposures at 600 and 750°C. This will result in a significant delay in this effort.

### **Plans for Next Quarter**

None; funding is exhausted.

## Task 3 Steamside Oxidation (B&W)

### Task 3A Autoclave Testing

#### Background

Steamside oxidation tests will be performed on commercially available and developmental materials at temperatures between 650°C and 800°C (1202°F - 1472°F).

#### Experimental

No laboratory testing was possible during the past quarter due to the relocation of the BWRC laboratory. Upon further review of the EDS results from 750°C, sulfur was observed in many of the cross sections (Figures 1-3). Thus, it appears that the test system was contaminated with sulfur during the 750°C tests. A different retort was used for the 750°C tests than was used for the 650°C and 800°C tests, so it is likely that the retort was the source of the sulfur contamination.

It is likely that the sulfur contamination was largely responsible for the somewhat anomalous test results that were described in the previous quarterly report (many materials exhibited higher than expected oxidation rates). The final 2,000 hour exposure at 750°C will not be performed on the remaining specimens, since they have been exposed to non-typical steamside chemistry conditions. Oxidation testing at 750°C in a non-sulfur contaminated retort will be performed in the future.

The following conclusions from the 750°C tests are still valid:

- 1) Mechanical surface treatments improve the steamside oxidation behavior. Deep rolling appears to be more beneficial than shot peening.
- 2) RA602CA has a low oxidation rate and forms a Cr oxide above an Al oxide.
- 3) Alloy 282 displays Al oxide grain boundary penetrations.
- 4) The matching weld metals for Alloys 740, 230 and CCA617 have the same steamside oxidation behavior as their base metals. All of the weld metals, like their respective base metals, also display Al oxide grain boundary penetrations.

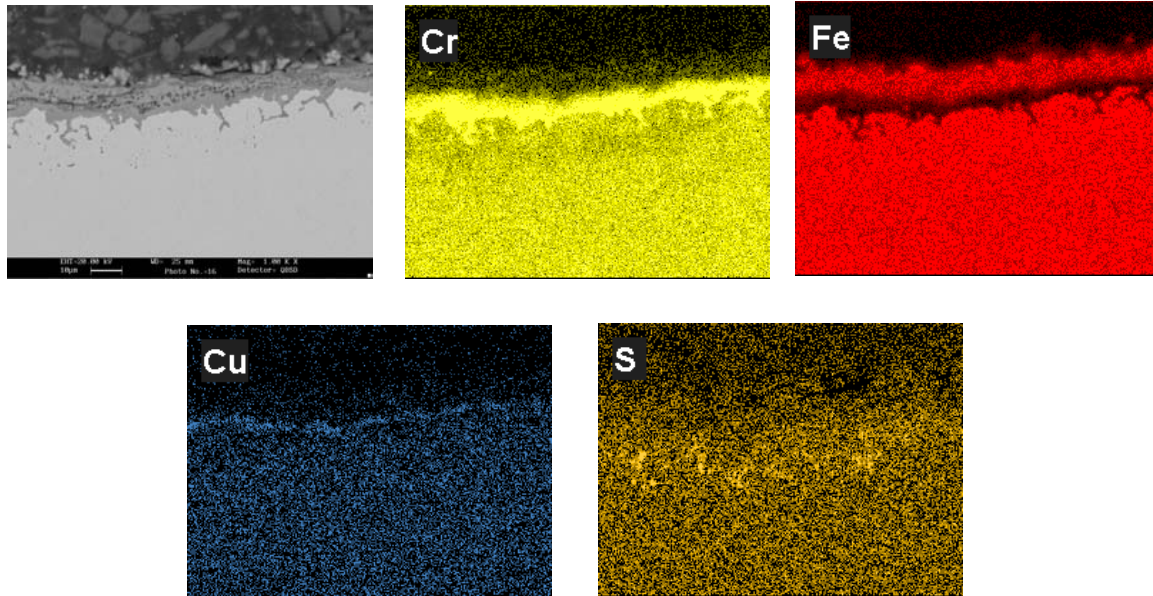


Figure 1. SEM/EDS of SUPER304H Tested at 750°C Showing Sulfur Penetrations

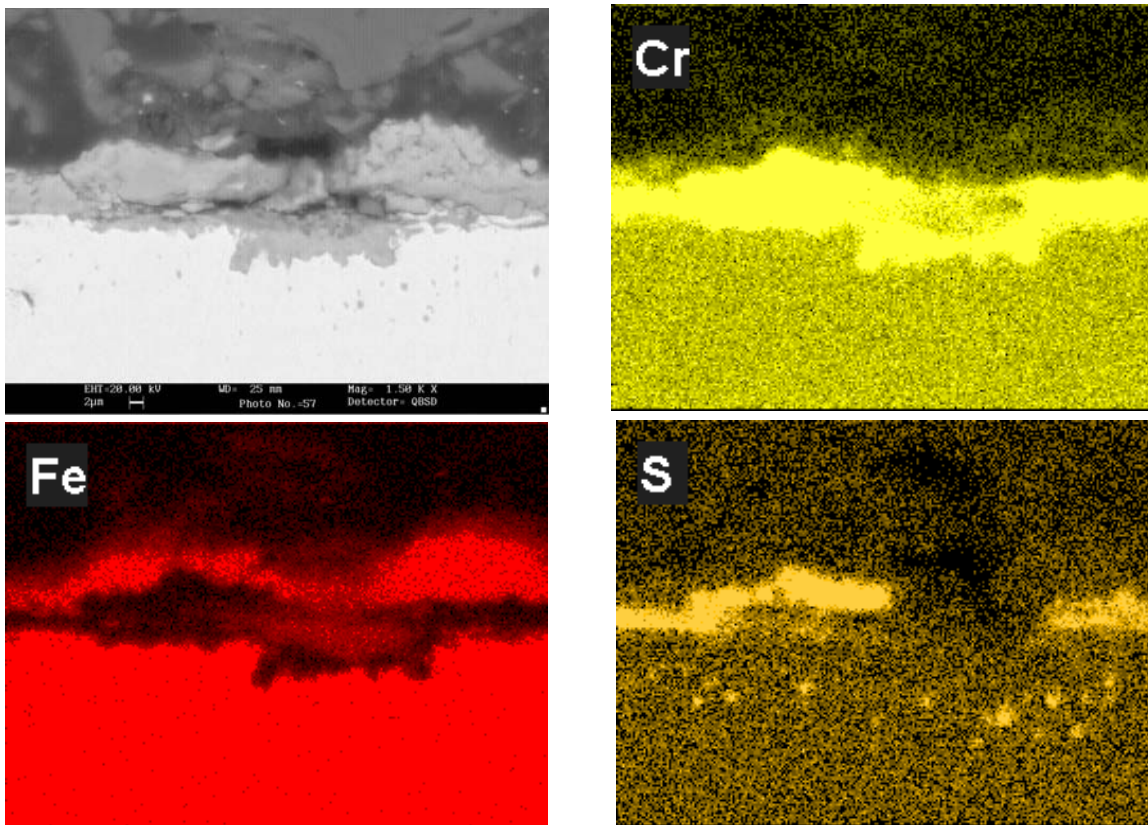


Figure 2. SEM/EDS of VM12 Tested at 750°C Showing Sulfur in Oxide Layers

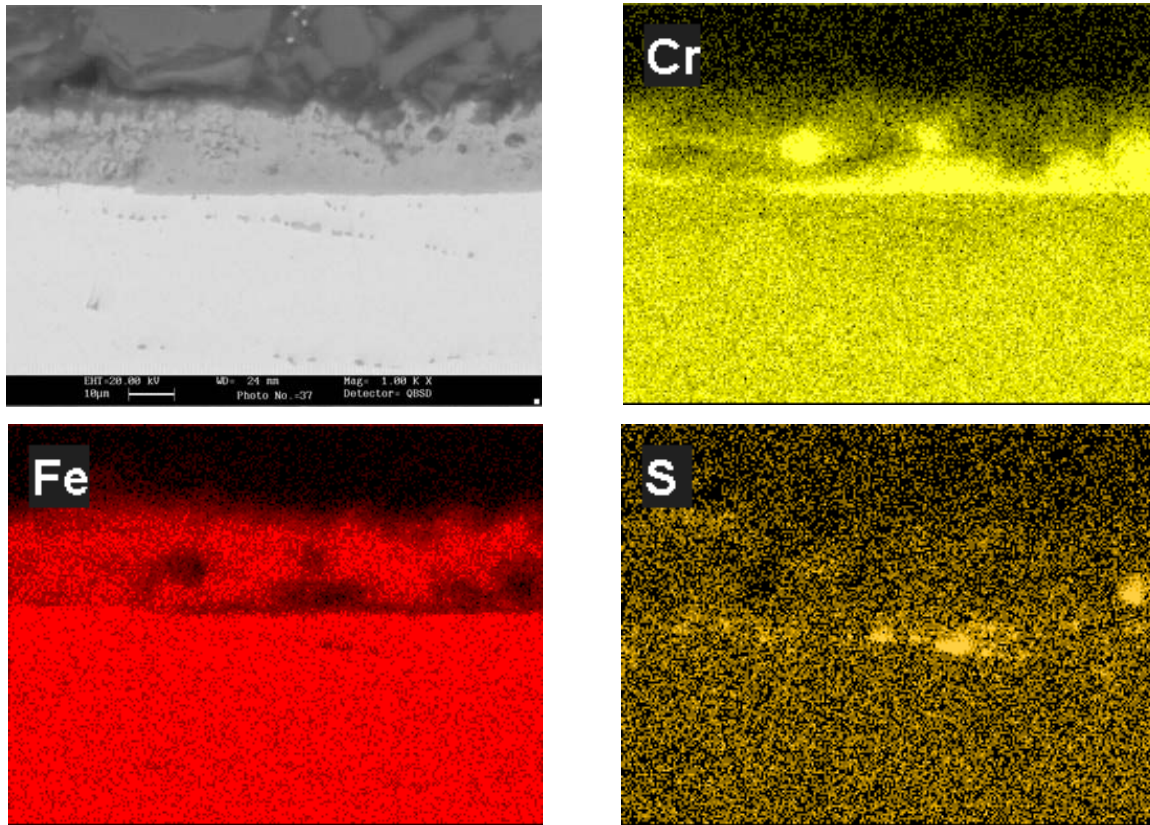


Figure 3. SEM/EDS of 304H Tested at 750°C Showing Sulfur Penetrations

Concerns

None.

Activities Next Quarter

Preparations will begin for the 750°C re-test.

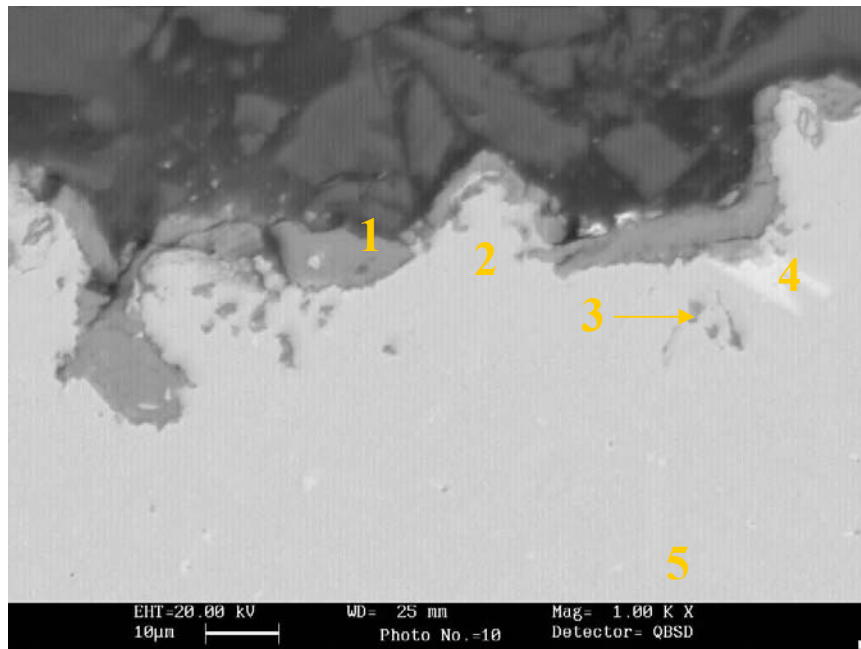
**Task 3B Coating Tests**

Background

Coated specimens for steamside oxidation testing will be prepared in conjunction with Task 7 and evaluated after testing.

Experimental

As with Task 3A, no experimental work was possible during the past quarter. The coated specimens tested at 750°C were exposed to the same sulfur-contaminated environment as the remainder of the non-coated specimens. As shown in Figure 4, some of these specimens also displayed evidence of the sulfur contamination.



	O	Si	S	Cr	Fe	Ni	Cu	Nb
sicrs304h-750-1-ba10 - area 1	22.5	0.4	0.2	72.0	3.5		1.5	
sicrs304h-750-1-ba10 - area 2		1.7		15.9	68.8	11.7	1.9	
sicrs304h-750-1-ba10 - area 3	10.8	2.6	0.6	31.8	45.1	7.8	1.3	
sicrs304h-750-1-ba10 - area 4		1.2		30.9	21.0	3.2		43.8
sicrs304h-750-1-ba10 - area 5		2.2		30.5	56.4	8.9	2.1	

Figure 4. SEM/EDS of 304H Tested at 750°C Showing Sulfur Penetrations

Concerns

None

Activities Next Quarter

It will be determined if additional coated specimens will be prepared for the 750°C re-test.



### **Task 3C      Assessment of Temperature**

#### Background

Based on the steamside oxidation test results, the practical temperature limits for the materials tested will be determined.

#### Experimental

As with Task 3A, no experimental work was possible during the past quarter.

#### Concerns

None

#### Activities Next Quarter

None

### **Task 3D      Review of Available Information & Reporting**

#### Background

Available steamside oxidation literature pertaining to materials and environmental conditions of interest will be reviewed. Project status updates will be prepared and status meetings will be attended as required.

#### Experimental

A presentation of Task 3 results to-date was prepared for a Steering Committee Meeting that will be held in Marco Island, FL in October, 2007.

A technical paper and presentation were prepared for the 5<sup>th</sup> International Conference on Advances in Materials Technology for Fossil Power Plants. The presentation will be made in Marco Island, FL in October, 2007.

#### Concerns

None.

### Activities Next Quarter

The Steering Committee meeting and Marco Island Conference will be attended and presentations will be made. A Quarterly Report will be written for October - December, 2007.

## **Task 3E Conduct Experimental Exposures**

### Background

The steam oxidation behavior of model Fe-Cr alloys will be evaluated.

### Experimental

B&W is remaining cognizant of the ORNL tests on these model alloys.

### Concerns

None.

### Activities Next Quarter

BWRC will maintain cognizance of ORNL results.

## **Task 3F Characterization**

### Background

Samples of the model Fe-Cr alloys fabricated in Task 3E will be characterized before and after steamside oxidation testing using metallographic and electron optic techniques.

### Experimental

None.

### Concerns

None.

Activities Next Quarter

None

**Task 3G Data Analysis and Coordination**

Background

The steamside oxidation results will be evaluated to determine the effects of material properties and environmental factors on oxidation behavior.

Experimental

No progress will be possible until the steamside oxidation tests have been completed.

Concerns

None.

Activities Next Quarter

None.

**Milestone Chart**

Dates are listed in GFY

## **4 Fireside Corrosion (Foster Wheeler)**

The objective of the task is to evaluate the relative resistance of various advanced alloys for waterwall and reheater/superheater construction to fireside corrosion over the full temperature range expected for the USC plant. The corrosive environment promoted by three different coals, representative of an eastern coal, a midwestern coal, and a western coal, will be evaluated.

### **Task 4A Laboratory Testing**

#### **Objective**

The objective of this sub-task is to perform laboratory corrosion tests on the candidate alloys. This will be accomplished by exposing metal coupons to various deposits and flue gases representative of three coals (e.g., eastern, midwestern, and western) at the range of temperatures expected for the USC plant.

#### **Experimental Progress**

During the quarter, work continued on writing of the final report.

#### **Concerns**

- No concerns at this time.

#### **Plans for Next Quarter**

- Finish some minor analytical work and introduce findings into the final report. The final report should be completed or near completion by the end of the quarter.

## Task 4B Corrosion Probe Testing in Utility Boilers

### Objective

The objective of this sub-task is to install corrosion probes comprised of various alloys/coatings/weld overlays at three coal-fired power plants and control them in the temperature range expected for reheater/superheater components in the USC plant. The plants should burn coals representative of the three types specified earlier. At the end of the testing, the corrosion probes will be removed and material specimens will be evaluated for corrosion resistance.

### Experimental Progress

- The first pair of corrosion probes was installed in host utility site burning Mid-Western coal during January 2006. The one-year probe was removed in April and laboratory evaluation of the samples commenced shortly thereafter. Preliminary results of the evaluation were presented at the Clearwater Conference in June 2007.
- The second pair of corrosion probes was installed at a utility host site burning Western coal during June 2006. The one-year probe was removed in late July and sent back to the laboratory for evaluation. Preliminary analysis has commenced and results were presented at the Marko Island Conference in October 2007.
  - Figure 1 shows a wide angle view of the entire probe surface after exposure to Western coal conditions. The probe was generally covered very thick, most poorly adherent ash deposits, the majority of which were dislodged from the surface during shipping and unpacking. The deposits appeared to be locally heaviest at the 12 and 4-5 o'clock positions, which was along the periphery of where the flue gas was impinging on the surface (about the 2 – 3 o'clock position).
  - Figures 2 – 6 are photomicrographs showing ring sections that were removed from coupon samples residing within a given temperature zone. Figure 2 shows materials removed from the hottest zone, Zone 1, while Figure 6 shows samples removed from the coolest zone, Zone 5.
  - Significant wastage was noted on most weld overlay/cladded coupon samples that contained >25% Cr and high Ni, regardless of the temperature. Several samples sustained localized metal loss of greater than 10%, relative to the original wall thickness.
  - For the most part, the wrought materials experienced shallow wastage, with the exception of HR6W and alloy 230 coupons removed from Zone 1. The penetration on both samples was intergranular.

- The third pair of corrosion probes (Eastern coal) has been built. Deployment of the third pair of probes is scheduled to occur in the Q4 2007 at an Ohio utility.
- Foster Wheeler continues to work closely with the host utility personnel to coordinate continuing exposures, maintenance tasks, and installation activities, as needed.

### **Concerns**

The original third (Eastern coal) host site has changed to 100% low-sulfur PRB (Western) coal. This required negotiations to secure a new host site. Negotiations have been successfully concluded with a host site within Ohio. The additional time and manpower effort required to reach agreement with a new Eastern coal host site has extended the schedule, and will likely result in additional costs for this task. This Ohio utility has already allowed us to install new penetrations for the probes, during a recent outage, and will permit installation of the final set of corrosion probes to proceed immediately after the next planned outage, scheduled for Spring 2007.

### **Plans for Next Quarter**

- Monitor ongoing exposures at the installed probe sites firing Mid-western and Western coals.
- Continue the evaluation of the one-year probes removed from the Mid-western and Western utilities.
- Deploy the corrosion probes at the third host site in Ohio (Eastern coal).

## **Task 4C Steam Loop Design, Construction, and Testing**

### **Objectives**

The objectives of this sub-task are to design, build, and test experimental USC steam loops that will operate in a commercial boiler at metal temperatures up to 1400°F. The elements of this subtask include the following:

- Design and construct two test loops using commercially-available, high-temperature corrosion-resistant alloys selected for the USC Plant.
- Install and operate the test loops at the Reliant Electric power plant, which is located in Niles, OH and is burning high sulfur Ohio coal.
- Test and monitor the relative performance of the USC tube alloys, coatings, claddings, and weld overlays, which comprise the test loops, for a period of 18 to 24 months.

### **Experimental Progress**

Both test loops were removed from the boiler at the end of May after approximately 3-1/2 years of service. The loops are currently at being evaluated.

### **Plans for Next Quarter**

- Continue evaluation of the test loop.

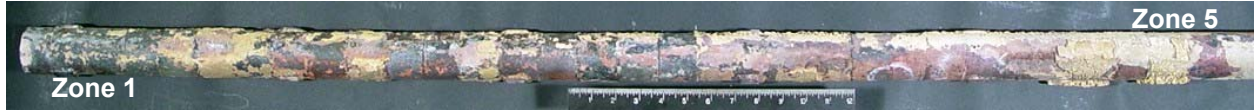


Figure 1: Corrosion Probe after one-year of exposure to Western coal conditions

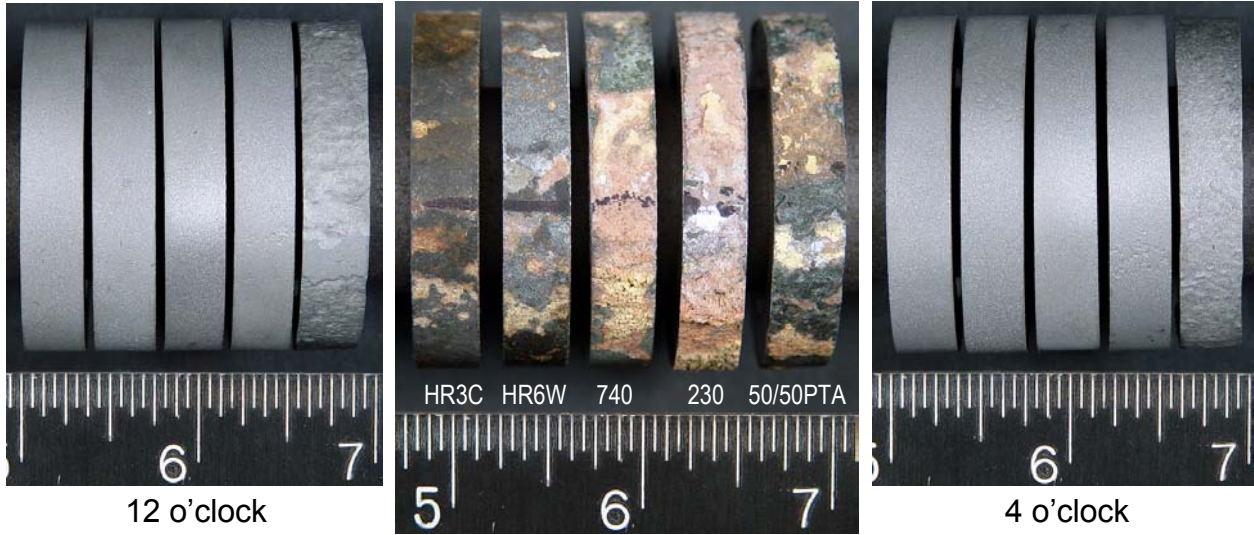


Figure 2: Close-up of rings sections of samples removed from Zone 1.

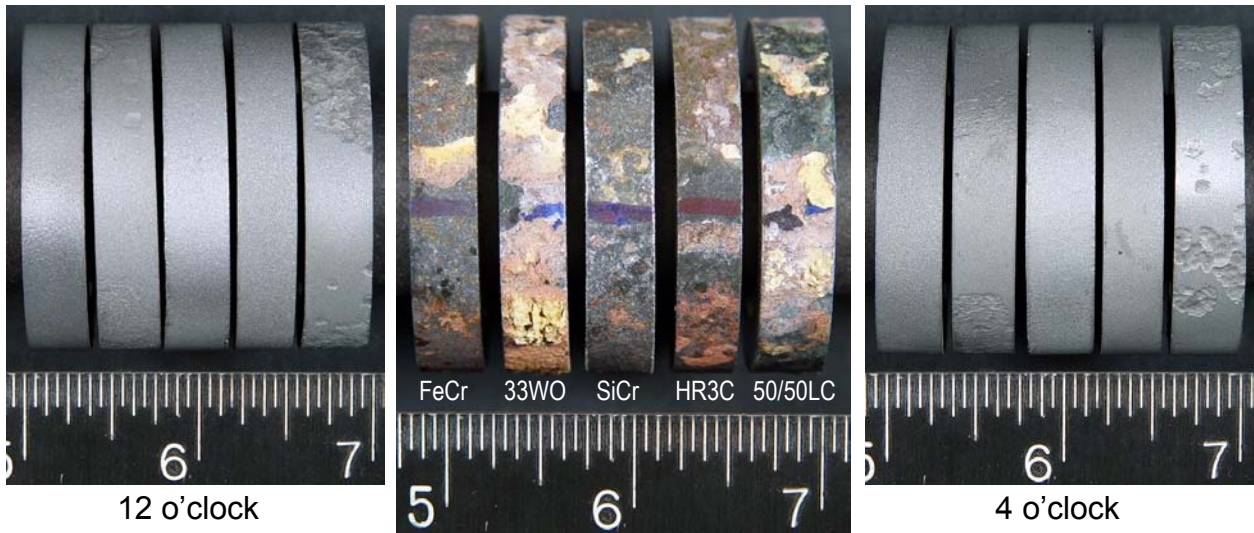


Figure 3: Close-up of rings sections of samples removed from Zone 2.



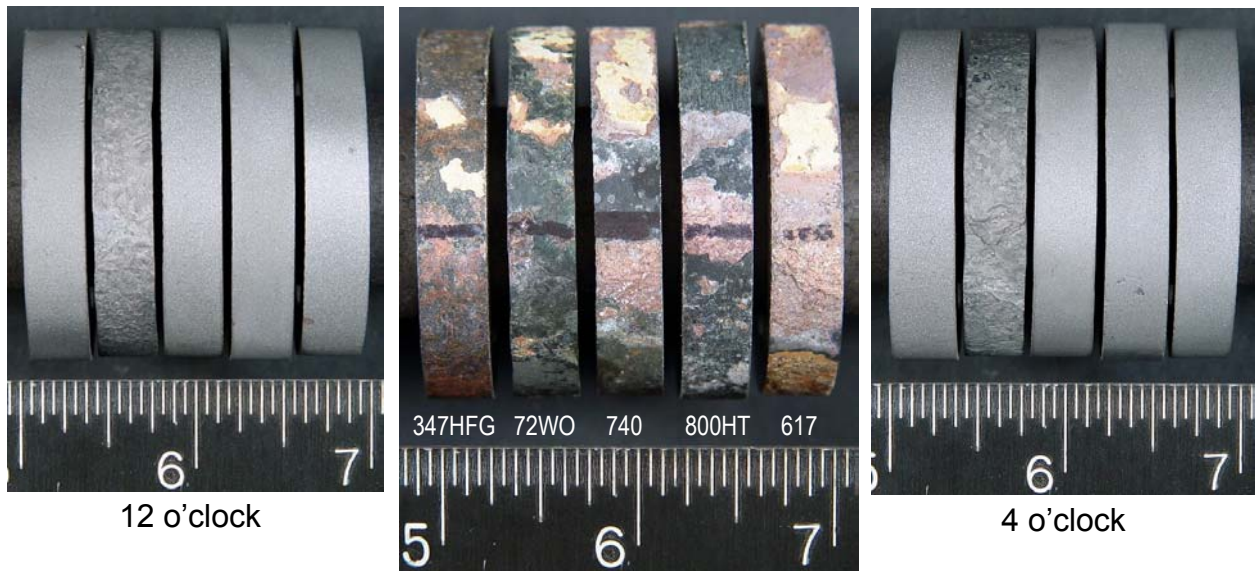


Figure 4: Close-up of rings sections of samples removed from Zone 3.

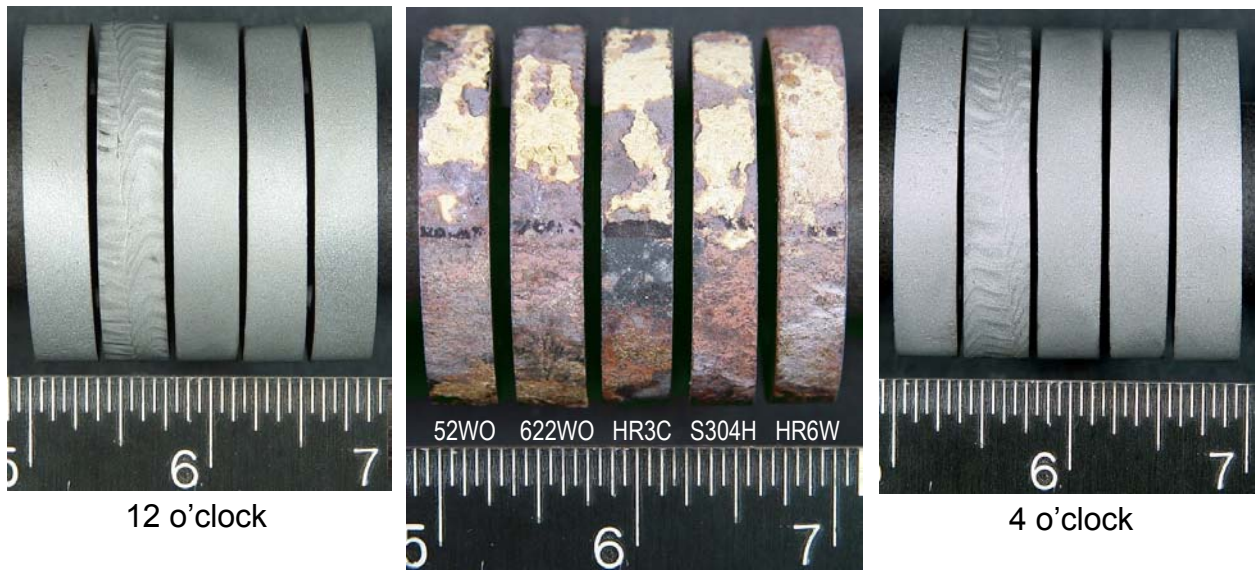


Figure 5: Close-up of rings sections of samples removed from Zone 4.

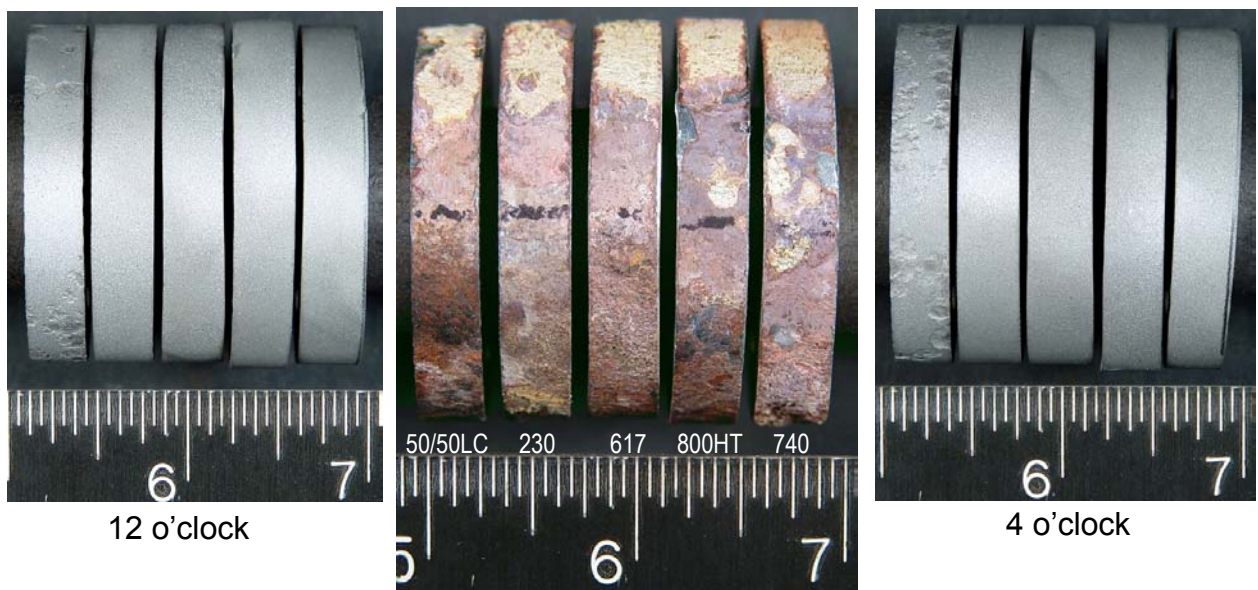


Figure 6: Close-up of rings sections of samples removed from Zone 5.