BOILER MATERIALS FOR ULTRASUPERCRITICAL COAL POWER PLANTS

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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_2 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 of 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_2 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 project is based on an R&D plan developed by the Electric Power Research Institute (EPRI) that supplements the recommendations of several DOE workshops on the subject of advanced materials, and DOE's Vision 21. 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 Company), Foster Wheeler and Babcock Power) has been developed.

C. Project Tasks

The project objective is expected to be achieved through 9 tasks as listed below:

Task 1.	Conceptual Design and Economic Analysis
Task 2.	Mechanical Properties of Advanced Alloys
Task 3.	Steamside Oxidation Resistance
Task 4.	Fireside Corrosion Resistance
Task 5.	Welding Development
Task 6.	Fabricability
Task 7:	Coatings
Task 8:	Design Data and Rules
Task 9:	Project Integration and Management

D. Major Accomplishments During the Quarter

- Procurement of IN 740, Haynes 230, Super 347H, HR6W, CCA617 and SAVE 12 has been completed.
- A GTAW procedure was qualified for welding thin Haynes 230 plates, Super 304H tubing and a GTAW root/SMAW fill procedure for Haynes 230 tubing.
- A review of state-of-the art for coatings was completed and a report issued.
- Design of the two USC test loops was completed. Weld overlay claddings (In 52, In 72, and In 622) of selected tubes that will be used to construct the loops (Inconel® 740, Hayes® alloy 230, Sumitomo HR6W, and CCA617) was completed.
- Cold U-bending and hot resizing fabrication trials with 2" OD x 0.400"MW alloy 230 tubing were conducted.
- A draft report on reference stress methodology and its application to ASME geometries was developed and issued.
- Steam oxidation testing of substrate and coated specimens was started.
- Planning and logistics for execution of the scale-up efforts for internal coating of tubing with pack cementation techniques continued.
- Creep testing of Super 304H, HR6W, alloy 230, and alloy 230 cross welds tubing has started.
- All creep-rupture tests on Inconel 740 bar were completed.
- A presentation on the design and testing plan for the USC test loops was presented to the management of Reliant Electric's Niles, OH facility. The test location within the boiler, which was down for maintenance on separate occasions during May and June, was inspected, measured, and photographed in preparation for USC loop installation.
- Trial #2 for the steam side oxidation testing was completed and the 650^{o}C test preparations were completed. A topical report on the literature review has been prepared, reviewed and finalized.

E. Plans for the Next Quarter

It is anticipated that the following work will be completed during the next quarter:

- Qualification of alternative Si-Cr pack cementation formulations. T-23, T-92 and S304H coupons will be used.
- Complete processing Super 304H coated tube samples for inclusion in the Niles steam test loop.
- Visit the First Energy's Berger station for a walk down and presentation in support of their being a host utility for corrosion probes.
- 37 of the 128 specimens from the steam oxidation test retort will be weighed and metallographically evaluated as they are removed from the vessel.

F. Issues

- SAW welding on Haynes 230 and Inconel 740 was not successful and efforts with this process on these materials will not continue.
- B&W discovered that the design allowable stresses for Alloy 740 material used for the conceptual design work under this Task need to be adjusted downward based on an evaluation of the latest information available from the alloy developer, Special Metals.

2.0 Taskwise Status

Task 2 Mechanical Properties of Advanced Alloys (ORNL)

The objective of Task 2 is to produce the database needed to design a boiler to operate at the steam conditions within the scope of the project.

Subtask 2A: Assessment of the Alloy Performance Requirements:

This assessment will focus on performance needed for boiler service in the temperature range of 649°C (1200°F) to 871°C (1600°F) and will provide justification for the materials selected for the pressure retention components of the boiler.

Drafts of parts I and II were completed last year. A new lead author has been appointed for Part III on stainless steels and high alloys.

Task 2B: Detailed Test Plan:

The mechanical properties test plan is being reviewed in light of the materials selected for the initial thrust of the project, and data are being collected for each of the alloys identified by the USC Project Team.

The creep rupture characterization testing matrices for Super 304H, HR6W, and alloy 230 have been developed. See the attached excel tables.

Task 2C: Long Term Creep Strength:

The objective of the long-term creep testing is to identify the general characteristics of the creep and damage accumulation in the candidate alloys.

Creep testing of Super 304H, HR6W, alloy 230, and alloy 230 cross welds tubing has started. Test numbers and conditions are included in the test plan matrices.

All creep-rupture tests on Inconel 740 bar were completed and an excel test summary table is provided in the attachments.

Task 2D: Microstructural Analysis:

The objective of the microstructural analysis is to identify the microstructural changes that significantly lead to strengthening, weakening, and internal damage characteristics of each material and to explore how these characteristics relate to the exposure conditions of the testing.

Specimens of alloy 230 and CCA 617 were sent to the University of Cincinnati for metallurgical characterization. Tubing samples were prepared for thermal aging.

Task 2E: Assessment of Creep-Fatigue Properties:

The objective of the creep-fatigue studies is to develop a database that will lead to practical yet conservative methods to address the issue of creep-fatigue damage in the boiler materials.

Repairs of the water cooling system are underway.

Task 2F: Modeling of Weld Joints:

The objective of Task 2F is to produce the experimental data needed to model dissimilar metal and thick section weld joints.

Specimen designs have been prepared for review by Task 8.

Task 2G: Study of Accelerated Testing Methods:

The objective of the accelerated testing is to provide a method to rapidly characterize changes in the strength of the candidate materials.

Checkout of an ATS relaxation testing device was completed. It was determined that the devices were not suitable for the relaxation tests that were intended.

An electro-hydraulic testing system was refurbished and operators trained to operate relaxation tests. Here, testing times will be restricted to 100 hours or less.

Task 2H: Model Validation:

The objective of the model validation testing is to produce a database that can be used to confirm or validate the design rules that are developed in Task 8.

Some specimen designs were drafted for review by Task 8.

Super 304H

TN	SN	Temperature (Deg. C)	Stress (MPa)	Expected Life (h)
		600	240	10000
		600	280	1000
		600	340	100
30293		650	120	10000
30292		650	210	1000
		650	260	100
		700	110	10000
30294		700	160	1000
		700	210	100

HR6W CREEP RUPTURE

TN	SN	Temperature (Deg. C)	Stress (MPa)	Expected Life (h)
30282		650	200	1000
		650	175	6000
		650	150	20000
		675	200	500
		675	170	1000
		675	150	6000
		700	200	50
		700	170	500
		700	150	1000
		700	120	10000
		725	170	50
30283		725	150	500
30291		725	120	5000
		750	140	100
		750	120	500
		750	100	5000
		750	85	20000
		775	100	600
		775	85	5000
		800	100	150
		800	85	1000

Alloy 230 CREEP RUPTURE

TN	SN	Temperature (Deg. C)	Stress (MPa)	Expected Life (h)
		650	350	200
30290		650	300	1000
		650	200	10000
		700	300	100
		700	200	1500
		700	140	15000
		750	200	100
		750	140	2000
		750	100	15000
		800	140	100
		800	100	2000
		800	80	20000

Inconel 740 bar

TN	SN	Condition	T (deg. F)	S (ksi)	mcr	RL (hours)
30184		Age	1450	25		3630
30196		Age	1500	17.5		3399
30183		Age	1500	20		2060
30106		Age	1400	30		5554
30011		Age	1500	20		2500
29993		1925F SA	1500	20		319.9

Age = 2204 F anneal + 16 hours at 1472F

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 900°C (1202°F - 1652°F).

Experimental

During this quarter, Trial #2 was completed and the 650°C Test preparations were completed. By the end of June, the 650°C Test had been initiated. These activities are discussed in detail below:

Trial #2

The basic objectives of Trial #2 was to insure that the test facility constructed for steamside oxidation testing will operate as expected, to determine proper set points prior to initiating the 650°C test, and to gain experience in chemistry preparation, specimen handling, etc.

Trial #2 of the steamside oxidation test facility for USC Task 3A was performed between April 2 and April 21, 2003 on 4 Alloy 188 (3.5% Fe, 22% Cr, 22% Ni, 14% W, Bal. Co) coupons and 4 T-11 (1.25% Cr, 0.55% Mo, Bal. Fe) coupons. The dimensions of all the coupons were ~1”x1/2”x1/4” thick. Following machining, the coupons were cleaned, dried, measured and weighed. The coupons were then placed in a ~16” long mullite specimen holder. To evaluate any front-to-back variations, 2 coupons from each material were placed in the front of the specimen holder, and two specimens from each material were placed in the rear of the specimen holder. The mullite specimen holder was placed in an Alloy 601 test rack which was positioned inside of the retort, which had already been positioned inside of a furnace.

Once the retort head was bolted onto the retort, a vacuum was drawn on the retort. The retort was then back-filled with argon gas. The retort was evacuated and back-filled with argon three times. The argon-filled retort was then heated to 650°C (1202°F). At the beginning of the heat-up, and throughout the remainder of the trial, a data acquisition system (DAS) collected temperature data from thermocouples (TCs) placed at three locations within a thermal well that was attached to the retort head and protruded into the retort. The TC locations were at the front of the test rack, just past the middle of the test rack, and at a location between the front and middle of the test rack. The thermal well is positioned near the top of the retort.

After the temperature in the retort was stable at 650°C, test solution (58 ppb NH_3 and 113 ppb dissolved O_2 in high purity water) was pumped through tubing in the furnace where it flashed to steam. The steam passed through the retort and was condensed to water outside of the furnace. The water was initially pumped at 8 ml/min. After ~1 day the flow was increased to 16 ml/min, and after another day the flow was increased to 24 ml/min. The flow was decreased to 8 ml/min and the temperature was increased to 800°C. The flow was again cycled through 8, 16 and 24 ml/min, with each flow being maintained for ~1 day. The temperature was then increased to 900°C and the flow was again cycled through 8, 16 and 24 ml/min, with each flow being maintained for ~1 day.

At the conclusion of the exposure, the furnace was cooled, the retort was opened and the specimens were removed. It was observed that the T-11 specimens had oxidized to such an extent that the oxide growth was impeded by the mullite test rack. The Alloy 188 specimens displayed a thin dark oxide. Examples of the post-test condition of the specimens are displayed in Figure 1.

Figure 1. Condition of Alloy 188 (Specimen 045) and T-11 (Specimen 049) at the Conclusion of Trial #2

The specimens were weighed. Half of the specimens were descaled and re-weighed, while the other half of the specimens were metallographically examined. The results from the post-test weights are displayed in Table 1.

Table 3
Oxidation Rates and Corrosion Rates for Trial #2 Specimens

Specimen #	Material	Location	Time Averaged Temperature (C)	Approximate Exposure Time (Days)	Weight Change per Unit Area (mg/cm^2)	Oxidation Rate (g/cm^2sec)	Descaled Corrosion Rate (mpy)
03-045	Alloy 188	Rear	797	19	0.000	1.996E-22	1.152
03-046	Alloy 188	Front	797	19	0.498	3.032E-10	
03-047	Alloy 188	Front	797	19	0.442	2.694E-10	0.876
03-048	Alloy 188	Rear	797	19	0.520	3.169E-10	
03-049	T-11	Rear	797	19	337.446	2.056E-07	1015.773
03-050	T-11	Front	797	19	302.479	1.843E-07	
03-051	T-11	Rear	797	19	316.750	1.930E-07	
03-052	T-11	Front	797	19	338.989	2.065E-07	993.123

The metallographic evaluation indicated that the scale on Alloy 188 was very thin. The scale on T-11 consisted of two layers, as shown in Figure 2.

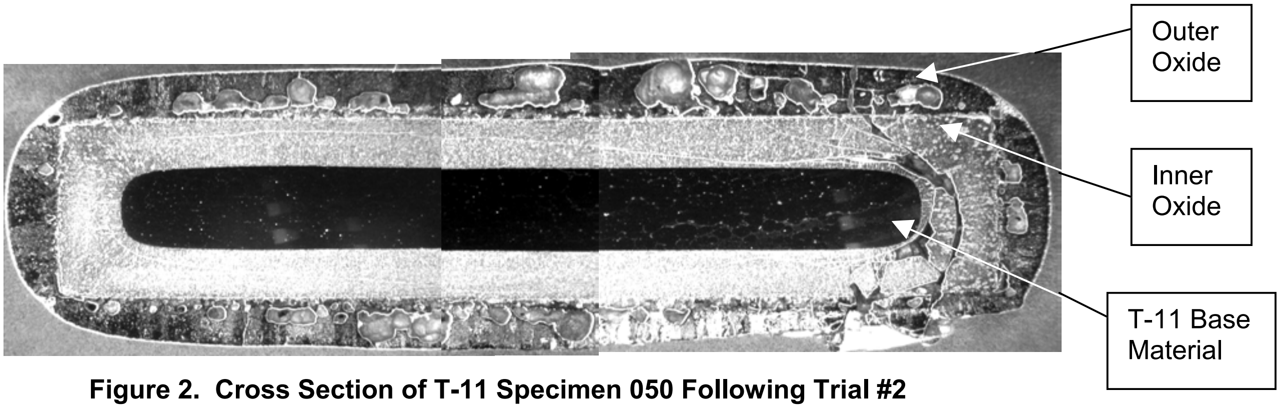


Figure 2. Cross Section of T-11 Specimen 050 Following Trial #2

The outer layer was quite porous while the inner layer was dense. The demarcation between these two layers appeared to be the original specimen surface, suggesting that the oxide grew both outward and inward.

During the test, oxygen concentration measurements were taken on the condensed retort effluent and indicated that oxygen was present in the effluent. These measurements along with the appearance of the specimens, and the weight change data all clearly indicate that the specimens were exposed to an oxygen containing environment. The visual observation of the specimens and the weight change data indicate that there are no significant front-to-back oxygen concentration variations within the retort.

For the T-11 specimens, the approximate calculated oxidation rate at the time averaged temperature of 797°C was $2x10^{-7}g/cm^2sec$. Figure 3 from CORROSION2002 Paper 02377 (by Ian Wright and Bruce Pint) indicates that ferritic alloys containing 0-2%Cr displayed an oxidation rate of $\sim1x10^{-7}g/cm^2sec$ (assuming linear kinetics and an activation energy of 157 kJ/mole). The agreement between the Trial #2 oxidation rate and the literature value, especially considering the number of different temperatures and flows evaluated in Trial #2, was quite good. As expected, the Alloy 188 exhibited very little oxidation in this trial.

While the steam oxidation results from the coupons are interesting, the most important finding from Trial #2 is that the test system performed very well at all temperatures and flow rates evaluated for ~500 hours. In the process, the furnace set points for each condition have been recorded to minimize temperature variation during subsequent tests.

A summary memo was issued containing the details of Trial #2.

650°C Test

During the past quarter, all the remaining test materials were received for the 650°C test. These materials are listed in Table 2.

Table 2
650°C Test Materials

P91	T23	Diffusion Chrome HR-120
P92	"9 Cr"	Diffusion Chrome RA253MA
Alloy 230	Nimonic 263	Diffusion Chrome RA333
HR-120	Alloy 800HT	Diffusion Chrome Alloy 617
SAVE 25	Chromized P92	Diffusion Chrome Alloy 740
CCA617	Silicon-Chrome P92	Diffusion Chrome RA353MA
Super 304H	Aluminum-Chrome P92	Diffusion Chrome Alloy 230
Alloy 740	Electroless Ni P92	"9 Cr" PreOxidized *
HR6W	Cereblak Coated P92	"9 Cr" Nano Nitrides *
SAVE 12	Diffusion Chrome RA602CA	VM12 *
304H SS		

* - Added after the first 1,000 hour exposure

As shown in Table 2, three additional materials were provided that will be placed into test after the first 1,000 hour exposure is complete.

Based on the results from Trial #2, it was decided to hang the specimens from the Alloy 601 test frame instead of setting the specimens in a mullite test rack. Thus 1/8" holes were drilled at the top of each specimen. After all the specimens were machined, drilled, cleaned, measured and weighed, they were attached to the test frame using Nichrome wire. Figure 3 shows the test frame with all of the 650°C specimens prior to the first 1,000 hour exposure.

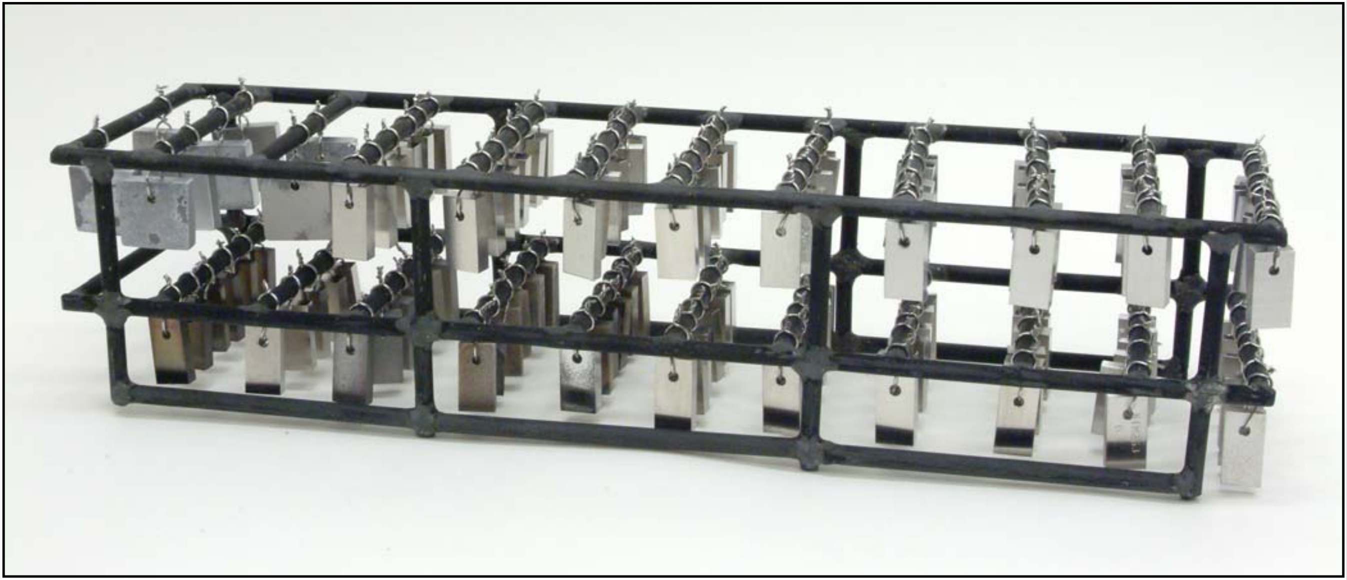


Figure 3. 650°C Test Frame with Specimens Pror to First 1,000 Hour Exposure

The test frame was placed into the retort which was sealed. The retort was sealed and evacuated and re-filled with argon three times before heating to ~650°C under argon. Once the temperature became stable at ~650°C, the test solution was pumped through tubing in the furnace where it flashes to steam and enters the retort.

Test conditions were reached on June 23. Temperatures and the dissolved oxygen content of the retort effluent are being continuously monitored on a data acquisition system (DAS).

- Concerns: There are no concerns at this time
- Activities Next Quarter: The 650°C Test will continue through the next quarter. It is anticipated that the first 1,000 hour exposure will conclude in early August. The second 1,000 hour exposure should conclude in late September or early October. Following each exposure, specimens will be removed, weighed and metallographically evaluated. The additional specimens that will be added to the test following the first 1,000 hour exposure will be prepared.

Task 3B Coating Tests

Background

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

Experimental

All of the coated specimens that were provided are currently being exposed in the 650°C Steamside Oxidation Test. A listing of the coated specimens is shown in Table 3.

Table 3
Coated Materials for the 650°C Test

Material	Number of Coupons	Source
Diffusion Cr on Alloy 617	1	B&W
Diffusion Cr on Alloy 740	1	B&W
Diffusion Cr on RA353MA	1	B&W
Diffusion Cr on Alloy 230	1	B&W
Diffusion Cr on RA253MA	1	B&W
Diffusion Cr on RA333	1	B&W
Diffusion Cr on RA602CA	1	B&W
Diffusion Cr on Alloy 120	1	B&W
Chromized P92	6	Alstom
Si-Cr on P92	6	Alstom
Al-Cr on P92	6	Alstom
Electroless Ni on P92	6	Alstom
Cerablak on P92	6	Alstom

- Concerns: There are no concerns at this time
- Activities Next Quarter: The coated materials will be weighed and metallographically evaluated as they are removed from the 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

No progress will be possible until results from the steamside oxidation tests are available.

- Concerns: There are no concerns at this time
- 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

The Literature Review was revised and submitted for review. EPRI re-organized the review and returned the Review to B&W for comments. The B&W comments were sent to EPRI.

Monthly status reports were prepared for April, May and June, 2003, a Quarterly Report was prepared for the January-March, 2003 time period, and a Quarterly Report was prepared for the April-June, 2003 time period.

- Concerns: There are no concerns at this time

- Activities Next Quarter: The literature review will be issued. Monthly status reports will be written for July, August and September, 2003.

Work will begin on the paper for the EPRI Materials and Corrosion Experience for Fossil Power Plants Conference in November in Charleston, SC.

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: There are no concerns at this time

- Activities Next Quarter: B&W will maintain cognizance of ORNL activities pertaining to model alloy test 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: There are no concerns at this time
- Activities Next Quarter: B&W will maintain cognizance of ORNL activities pertaining to model alloy characterization.

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 (GFY2006).

- Concerns: There are no concerns at this time
- Activities Next Quarter: None.

Task B: Steamside Corrosion Resistance (ORNL)

Corrosion coupon testing are continuing in the “Kaiser rig” for exposure at 700°C and 250 psi.

(1) The initial sequential run in steam at 1 atm. pressure was aborted because a crack was discovered in the reaction tube, resulting in exposure to steam+ air for an unspecified time. The specimens are being examined for comparison of the scale morphologies with those from air-10% water vapor exposures in another program, to decide if there is salvageable information.

(2) Results so far from specimens run at 700°C (1292°F) and 1.7 MPa (250 psig) steam in a rig that became available to the program. The specimens are hung by Pt wire from alumina arms on an alumina center post, as shown in Fig. 1. The assembly is enclosed in a Haynes 230 reaction tube that is externally heated. The test is being run in increments of

500h, with the specimens removed and reweighed each time. Four samples of each alloy were initially exposed; one sample of each was removed for metallographic examination after 1000h.

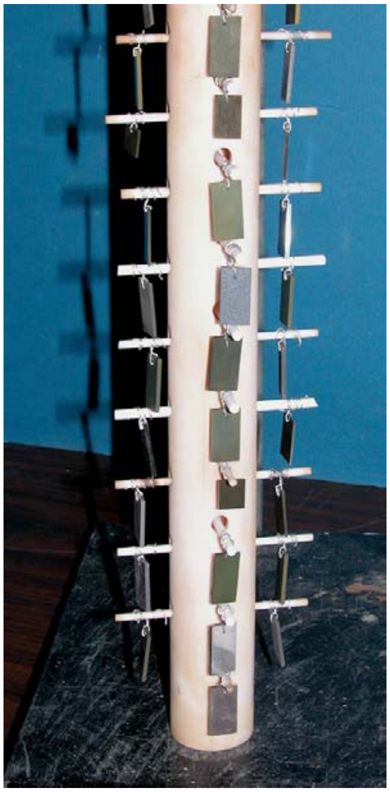


Figure 1. Specimen assembly removed from the reaction tube

The kinetics derived so far from mass change measurements are shown in Figs. 2 and 3. Note that direct comparisons, in the absence of metallographic data, are difficult, since the mass changes include contributions from scale spallation and chromium evaporation. Note also that not all the alloys scheduled for exposure are included, since they were not available when the tests were started. As these become available they are added to the test.

The relative mass changes show in Fig. 2 for T-22 and T-91 were unexpected and counter-intuitive. The possibilities are that:

(i) the alloy identities have been transposed; this will be addressed by electron -probe microanalysis of the specimens removed at 1000h.
(ii) T-22 suffered significantly more scale loss from spallation: differences between the alloy surfaces are difficult to discern; this will be addressed by metallography.

For the higher alloys (Fig. 3), the overall mass gains are, as expected, significantly lower than fort-22 and T-91. HR120 (25%Cr) and SAVE 25 (23%Cr) appear to yield consistently lower mass gains, but the relative tendencies for scale spallation have not yet been determined.

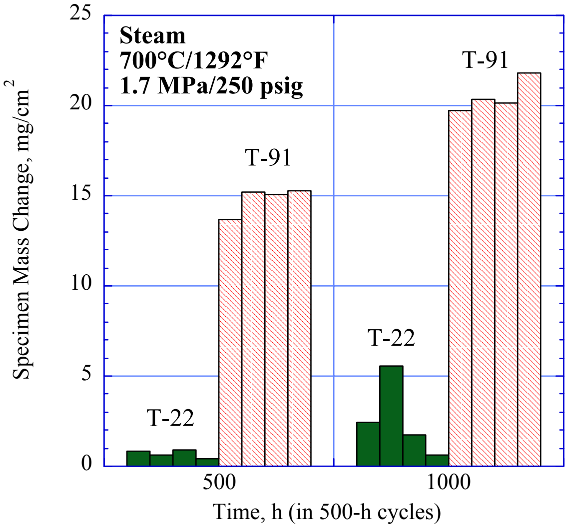


Figure 2. Mass-gain kinetics for low-Cr alloys

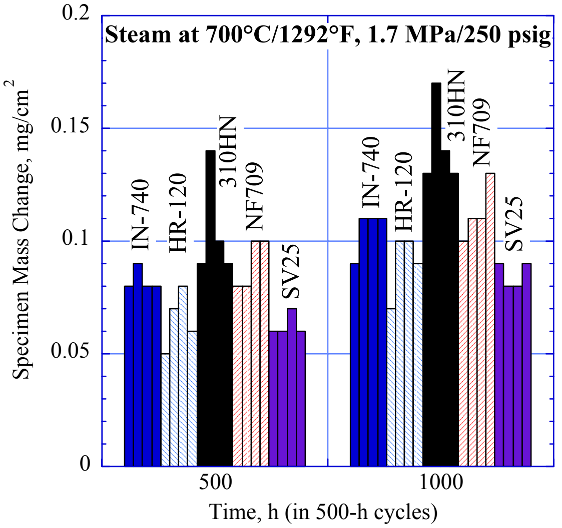
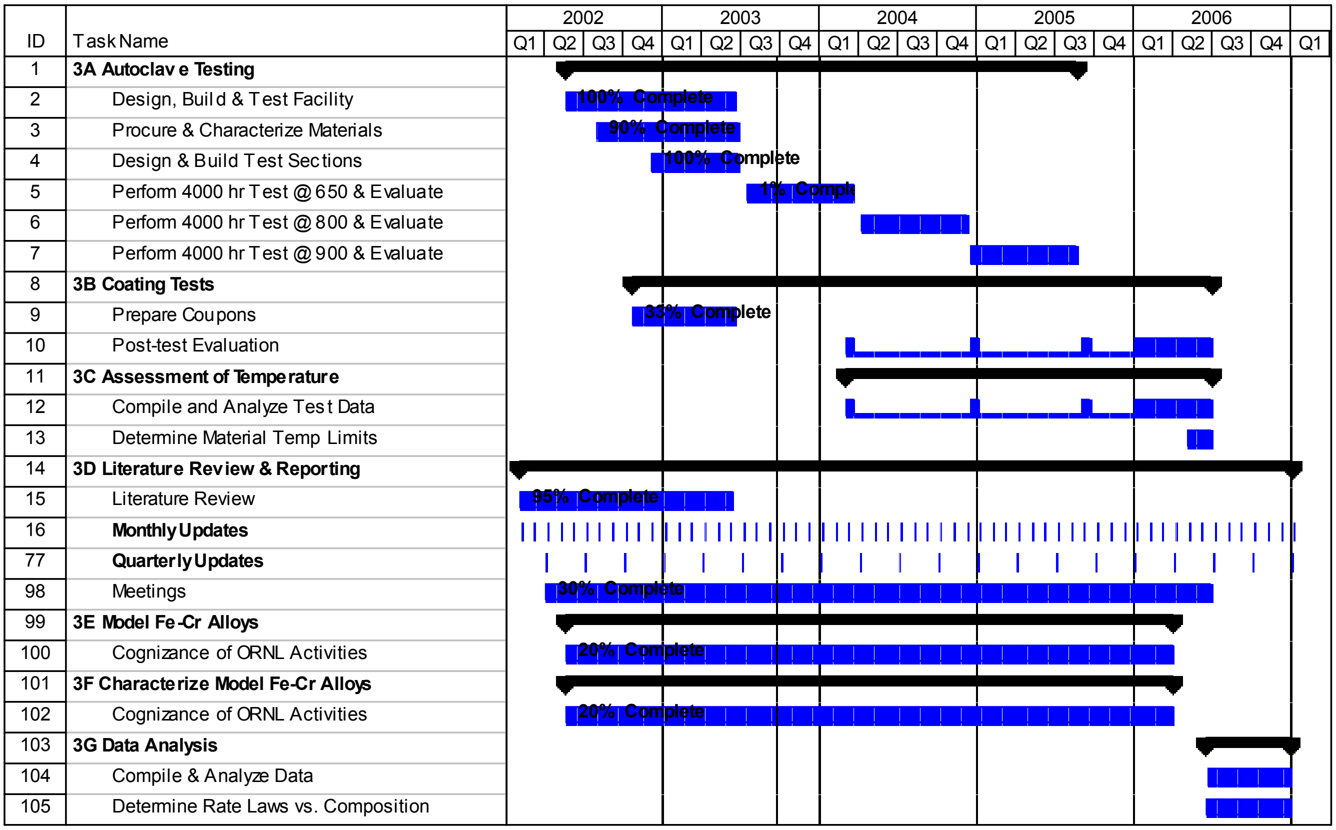


Figure 3. Mass gain kinetics for high-Cr alloys

Milestone Chart Task 3



ID	Task Name
1	**3A Autoclave Testing**
2	Design, Build & Test Facility
3	Procure & Characterize Materials
4	Design & Build Test Sections
5	Perform 4000 hr Test @ 650 & Evaluate
6	Perform 4000 hr Test @ 800 & Evaluate
7	Perform 4000 hr Test @ 900 & Evaluate
8	**3B Coating Tests**
9	Prepare Coupons
10	Post-test Evaluation
11	**3C Assessment of Temperature**
12	Compile and Analyze Test Data
13	Determine Material Temp Limits
14	**3D Literature Review & Reporting**
15	Literature Review
16	**Monthly Updates**
77	**Quarterly Updates**
98	Meetings
99	**3E Model Fe-Cr Alloys**
100	Cognizance of ORNL Activities
101	**3F Characterize Model Fe-Cr Alloys**
102	Cognizance of ORNL Activities
103	**3G Data Analysis**
104	Compile & Analyze Data
105	Determine Rate Laws vs. Composition

Task 4 Fireside Corrosion (Task lead Foster wheeler)

- The objective of the task is to evaluate the relative resistance of various advanced alloys to fireside corrosion over the full temperature range expected for the USC plant

Task 4A: Laboratory Testing

  - Objectives: To perform laboratory tests on candidate alloys exposed to various deposits representative of the three coals at the range of temperatures expected for the USC plant.

  - Progress for the Quarter: The assembly and checkout of the superheater test apparatus is continuing.

- Concerns: None

- Plans for the Next Quarter: Complete the procurement of the test materials and begin testing.

Task 4B: Corrosion Probe Testing in Utility Boilers

- Objective: To install corrosion probes of various alloys at three coal-fired power plants and control them at the temperature ranges expected for the USC plant.

- Progress for the Quarter: Previous corrosion probe designs are being reviewed in light of the higher test temperatures to determine the required probe dimensions. This information will be utilized for probe material procurement. Calls were made to follow-up on the Host solicitations.

- Concerns: No concerns at this time

- Plans for the Next quarter: Visit the FirstEnergy's Berger station for a walk down presentation on their being a Host utility

Task 4C: Steam Loop Design, Construction, and Testing (B&W)

- Objectives: The objectives of this subtask are to design, build, and test two experimental USC steam loops that will operate in a commercial boiler at metal temperatures up to 1400ºF. 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 Boiler Development Project.

- Install and operate the test loops at the Reliant Electric power plant located in Niles, OH, burning high sulfur Ohio coal.

- Test and monitor the relative performance of the USC tube alloys, coatings, claddings, and welds which comprise the test loops for a period of 18 months.

- Progress for the Quarter:

    - A presentation on the design and testing plan for the USC test loops was presented to the management of Reliant Electric's Niles, OH facility.

    - An information packet with *design allowables* for the materials to be used in building the test loops was completed and distributed to Task 4C participants. A separate information packet providing thermal expansion plots of USC alloys (from 200ºF to 1600ºF) was also prepared and distributed.

    - The design of the test loops was completed.

    - Tube section machining (ID sizing and weld end preparation) for loop construction was initiated.

  - First time weld overlay cladding of Inconel® 740, Haynes® alloy 230, CCA617 and HR6W tubes with Inconel® 52, Inconel® 72, and Inconel® 622 was successfully completed at Welding Services, Inc. in Marietta, GA.

  - Machining of both unclad tube sections (including Super 304H, RA333, Inconel® 740, Haynes® alloy 230, CCA617 and HR6W and clad tube sections, which will be butt welded to construct the high temperature legs of the test loops, was initiated.

  - Compilation of test loop documentation, including inspection reports, material test reports, and system test reports required by the Authorized Inspector (AI), was initiated.

  - The test location within the Reliant Electric utility boiler, which was down for maintenance on separate occasions during May and June, was inspected, measured, and photographed in preparation for USC loop installation.

- Concerns: None

- Plans for the Next Quarter:

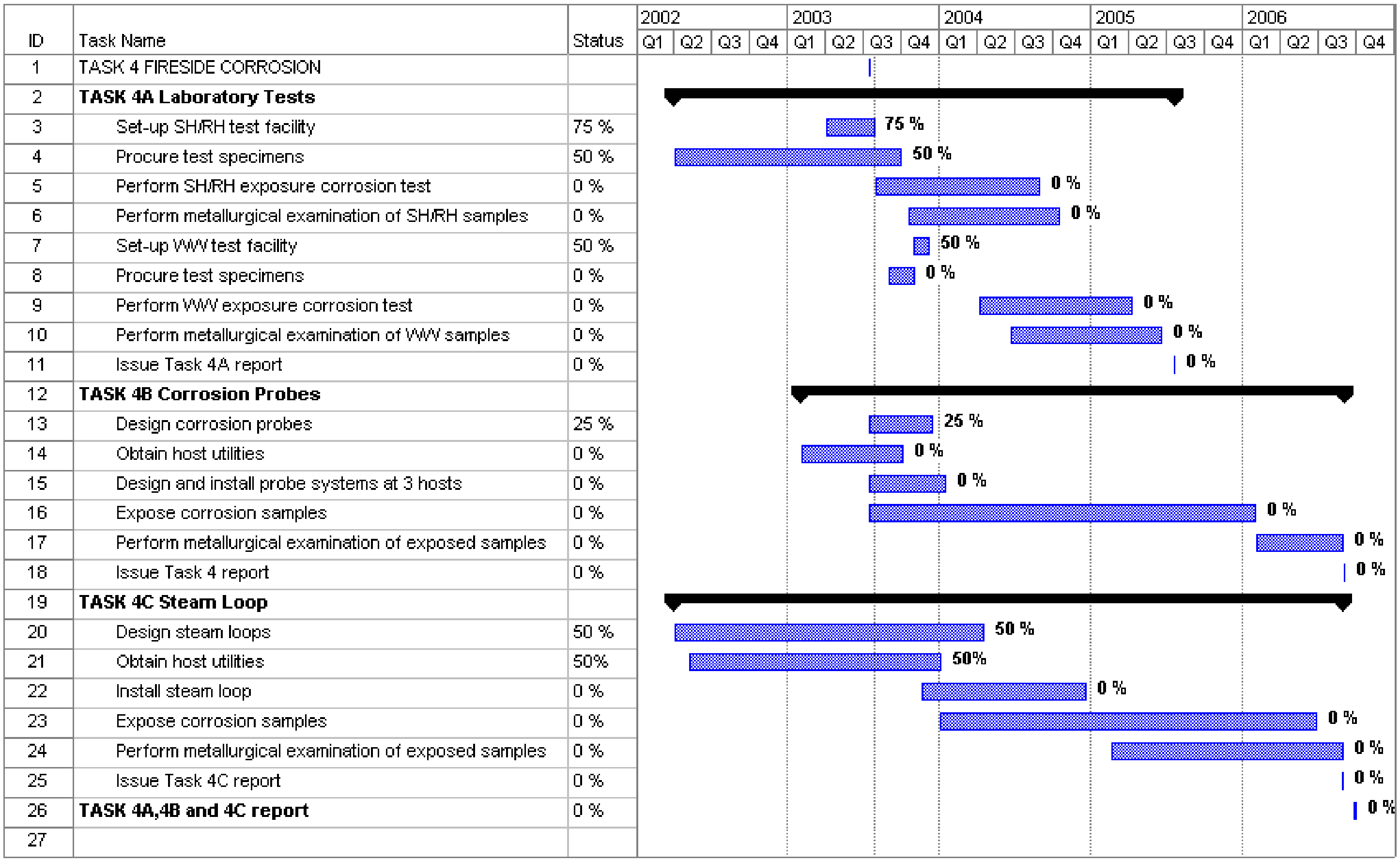
  - Machining, welding, assembly and inspection of the two test loop subsections will be completed at B&W Research Center in Alliance, OH.

  - All loop welds will be dye penetrant inspected and radiographically inspected, and the completed test loops will be hydrotested.

  - Subcomponents needed for the test loop assemblies (e.g., a specially fabricated attemperator, 310H tubing, steam throttling valves, thermocouples, etc.) will be procured and installed, along with the USC test loops at the Reliant Electric power plant in Niles, OH.

  - All required documentation for the test loops will be assembled and presented for inspection by the Authorized Inspector (AI).

Milestone Chart Task 4



ID	Task Name	Status
1	TASK 4 FIRESIDE CORROSION	
2	**TASK 4A Laboratory Tests**	
3	Set-up SH/RH test facility	75 %
4	Procure test specimens	50 %
5	Perform SH/RH exposure corrosion test	0 %
6	Perform metallurgical examination of SH/RH samples	0 %
7	Set-up WW test facility	50 %
8	Procure test specimens	0 %
9	Perform WW exposure corrosion test	0 %
10	Perform metallurgical examination of WW samples	0 %
11	Issue Task 4A report	0 %
12	**TASK 4B Corrosion Probes**	
13	Design corrosion probes	25 %
14	Obtain host utilities	0 %
15	Design and install probe systems at 3 hosts	0 %
16	Expose corrosion samples	0 %
17	Perform metallurgical examination of exposed samples	0 %
18	Issue Task 4 report	0 %
19	**TASK 4C Steam Loop**	
20	Design steam loops	50 %
21	Obtain host utilities	50%
22	Install steam loop	0 %
23	Expose corrosion samples	0 %
24	Perform metallurgical examination of exposed samples	0 %
25	Issue Task 4C report	0 %
26	**TASK 4A,4B and 4C report**	0 %
27		

Task 5 Welding Development (Alstom)

- To define weld metal choices for candidate materials.
- To evaluate weldability issues.
- To establish acceptable welding procedures and practices.
- To evaluate the effects of manufacturing heat treatments and preheat and post weld heat treatments on weldment integrity and properties.
- To produce samples needed to determine the properties of candidate ultrasupercritical alloy welds and weldments, including the dissimilar metal weld joint between the various types of material (actual mechanical and property testing will be performed under Task 2).
- Progress for the Quarter: The Task 5 efforts were directed towards procurement of the materials that will be used in the program, continued investigations of two of the nickel

base alloys, and initial studies of an austenitic stainless steel and another nickel base material. The individual procurement efforts include sufficient material to satisfy the requirements of all the various tasks.

With regard to each specific material involved in the welding studies:

- Super 304H.
  - All base material (tubing) and filler metals that were ordered have been delivered.
  - Attempts to weld Super 304H tubing using an automatic gas metal arc process were unsuccessful because the filler metal would not wet and tie into the base material, even with adjustments to travel speed and gas mixture. The basic process was that used for Type 347H tubing and a switch to Type 347 filler wire produced completely acceptable welds in the Super 304H tubing. Assistance is being sought from Sumitomo, the material supplier, and from other organizations that have had experience welding this material with a gas metal arc process.
  - An orbital gas metal arc process was successful in making tube butt joints and the qualification details for this process are being completed.

- CCA 617 (known in Europe as Marcko).
  - All base materials (tubing and plate) and filler metals that were ordered have been delivered.
  - Attempts to weld CCA 617 plate using a shielded metal arc welding process were unsuccessful because of poor weldability exhibited by the CCA 617 electrodes. Conventional Inconel 617 electrodes, which were procured and tested, had good weldability and did not exhibit the slag control problems that plagued the CCA 617 filler material. The chemistry of the various materials involved is being studied and suppliers are being contacted in attempts to understand the problem.
  - Attempts to make tube butt joints using an orbital gas metal arc process have been unsuccessful because of wire feed problems. The filler wire that was supplied is unusually stiff and has a larger diameter than is normally used for this process. These conditions cause the feed wire mechanism to be damaged shortly after welding is started. The initial observations indicate that the welding will be satisfactory once the feed problems are solved.
  - Equipment for submerged arc welding of the plate material has been set up and appropriate values for the various parameters are being established.

- Haynes 230
  - -- All required base materials and weld consumables have been procured.
  - -- A procedure qualification was successfully completed for manual welding of tubing with a gas tungsten arc root bead and shielded metal arc fill passes. The bend and tensile test specimens showed no evidence of welding defects or material related indications. Repair welds were also successfully simulated (based on radiography) on a tube to tube butt weld.
  - -- Several variants of the gas metal arc process were tried in order to develop a procedure for joining thick plates. Bead-on-plate welds using an automatic pulsed process displayed some microfissuring upon magnified visual inspection. Semiautomatic version welds did not contain this microfissuring, which actually appeared to be in the heat affected zone and not the weld metal. Experts in nickel alloy welding have seen this phenomenon before and the prime theory regarding its cause is liquation cracking from carbide or low melting point constituent formation. Because the cracking appears to be related to heat input, several additional attempts to eliminate the problem were made using manual gas tungsten arc, manual gas metal arc, and both high and low heat input automatic gas metal arc processes. Specimens consisted of 1/4-inch buildups from which side bend tests were extracted. Of all processes tried, the high heat input automatic gas metal arc weld showed no defects in the side bends. The other processes produced acceptable results with minor indications. A 1-inch thick groove weld is being prepared for trial using the high heat input automatic gas metal arc process.
  - -- All efforts to use a submerged arc process have resulted in cracking in side bend specimens regardless of the flux used. Therefore, this process will not be considered for thick section welds on Haynes 230.

- Inconel 740
  - -- Tubes were received and clad at Welding Services, Inc. with Inconel 622, Inconel 52, and Inconel 72 for use in the Niles Station reheater loops. All cladding went well and only the Inconel 72 filler required parameter adjustment. Compared to the others, the Inconel 72 was more difficult to use and had a smaller parameter operating window.
  - -- Initial development welds using a gas tungsten arc process were made on thin (0.250-inch) plate using Nimonic 263 filler. Parameter refinement was completed and the thin plate welding process is now ready for qualification once the Inconel 740 weld wire arrives. Repeated status requests for this wire were made to Special Metals and latest information indicated that the wire is now being drawn down and should be available in August.
  - -- All tests using potential submerged arc fluxes have been completed with negative results and the process has been eliminated from future weld trials.

    - -- Additional thick Inconel 740 plate that could be used on the proposed "follow on" weld development program was placed on hold by Special

Metals. This will assure that material is available if the workscope is approved.

- HR6W and SVE 12
  - -- All of the HR6W tubing and SAVE 12 pipe have been delivered and appropriate distributions have been made to other consortium members.
  - -- Program details are being developed for the selection of weld filler materials, the optimization of the welding procedures, preparation of sample material for laboratory testing, weldability testing, and the examination of dissimilar metal welds.

Concerns:

- Base material sourcing difficulties and long delivery times have, in some cases, delayed the start of welding activities by 9 to 12 months.
- The unexpectedly high cost of the nickel base alloys will cause the material budgets to be exceeded and might result in program cost overruns and/or reductions in program scope.
- Submerged arc welding, a high deposition rate process favored by boiler makers for thick sections, does not appear feasible for nickel base materials. Tests on Haynes 230 and Inconel 740 have been unsuccessful because of cracking and the process is being abandoned on these two alloys.
- Delivery of Inconel 740 weld wire continues to be a problem. Special Metals is now quoting delivery in August and any more slippage in schedule will impact delivery schedules.

Activities Planned for Next Quarter:

Plans for next quarter include:

- Build Niles test loops.
- Weld thin plate Inconel 740 (if wire is available).
- Begin thick plate welding of Haynes 230.
- Begin thick plate welding of Inconel 740.
- Fabricate Super 304H test specimens using gas tungsten arc process.
- Resolve issues with gas metal arc welding of Super 304H.
- Qualify gas tungsten arc process for CCA 617 tubing and fabricate test specimens.
- Resolve issues with shielded metal arc welding of CCA 617 plate.
- Begin submerged arc welding trials on CCA 617 plate.
- Plan details of welding efforts on HR6W and SAVE 12.

Task 5: Welding Development - Milestone Chart (DOE Fiscal Year Basis) (percentages indicate fraction of workscope completed as of 2003Q3)

		Year 2002				Year 2003				Year 2004				Year 2005				Year 2006			
Task	**Milestone**	**1**	**2**	**3**	**4**	**1**	**2**	**3**	**4**	**1**	**2**	**3**	**4**	**1**	**2**	**3**	**4**	**1**	**2**	**3**	**4**
5A	Selection of Weld Filler Material																				
	• Procure base metal for weld trials.	△					100%	▲													
	• Evaluation and selection of filler.	△					70%		▲												
	• Procurement of candidate fillers.	△					70%		▲												
5B	Optimization of Welding Parameters																				
	• Preliminary weld trials and parameter optimization – thin section.				△		30%					▲									
	• Preliminary weld trials and parameter optimization – thick section.				△		20%					▲									
5C	Preparation of Laboratory Samples																				
	• Material preparation.					△		5%					▲								
	• Sample fabrication.						△	5%					▲								
5D	Weldability Testing						0%	△						▲							
5E	Examination of Dissimilar Metal Welds.																				
	• Weld trials						0%				△			▲							
	• Metallurgical analysis						0%					△				▲					
	• Analysis and test case.																				

Task 6: Fabricability (B&W)

Task 6: Fabrication

- Objectives: The objectives of this task are to establish fabrication guidelines for the high temperature, corrosion resistant alloys selected for the USC Project. The elements of this task include the following:
  - To determine the effect of fabrication operations on the properties of USC alloys.
  - To determine the thermomechanical treatments or other remedial actions necessary to restore material properties which might degrade due to fabrication operations.
  - To investigate prototypical manufacturing operations for producing both thick wall and thin wall components from the USC alloys.
- Progress for the Quarter:
  - Foster Wheeler successfully demonstrated (and validated) a method to effect controlled straining of tapered tensile bars (demonstrated in both 304H and alloy 230) and a tapered tube specimen (demonstrated with 304H) to produce materials with different levels of strain necessary for characterizing the strain response, crystallization behavior, and precipitation behavior of USC materials.
  - As an incidental part of preparing tube sections to construct test loops under Task 4C, data on cutting, boring, lathe turning, weld overlay cladding, and butt welding has been accumulated for Inconel® 740, alloy 230, HR6W, Super 304H, RA333, and CCA617.
  - Cold U-bending and hot resizing fabrication trials with 2" OD X 0.400MW alloy 230 tubing were conducted at a B&W production facility. For cold U-bending, bend radii of 3", 5", and 7-1/2" were successfully made. For hot resizing, starting with 3" cold U-bends, radii of 2-1/4" were successfully produced.
- Concerns: None
- Plans for the Next Quarter:
  - Tapered tube specimens of USC alloys will be machined and control-strained to provide materials for characterization, thermal treatment and microstructural analysis. Some Task 6 participants may elect to subcontract some or all of this work to Foster Wheeler.

- Tube bending trials of other USC alloys will be conducted in commercial boiler production facilities.

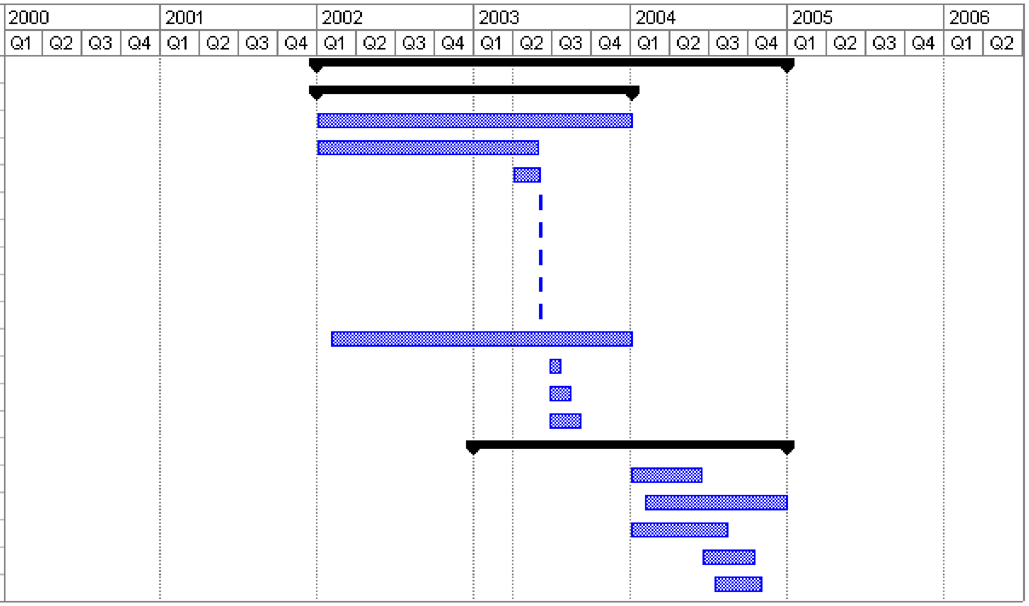
- Cold U-bend tube samples will be sectioned and metallurgically analyzed to characterize microstructures resulting from bending.

- Selected cold U-bend tube samples will be sent to ORNL for fatigue testing.

- A preliminary design of a fabrication demonstration article (incorporating all USC alloys, multiple types of machining operations, tube and plate bends, and welds) will be completed, and fabrication of this article will be initiated.

Milestone Chart Task 6

ID	Task Name	Start	Finish	Status
1	**Task 6: Fabricability**	Wed 1/2/02	Fri 12/31/04	In Progress
2	**6A: Fab Trials for SH**	Wed 1/2/02	Fri 1/2/04	Rev Pend'g
3	6A.1 SH Trial	Wed 1/2/02	Fri 1/2/04	Rev Pend'g
4	Procure Materials	Wed 1/2/02	Fri 5/30/03	Rev Pend'g
5	Shop Sched & Graphics	Tue 4/1/03	Mon 6/2/03	Rev Pend'g
6	Travel to Shop	Mon 6/2/03	Fri 6/6/03	Rev Pend'g
7	Cold Bending Trials	Mon 6/2/03	Fri 6/6/03	Rev Pend'g
8	Cold Swaging Trials	Mon 6/2/03	Fri 6/6/03	Rev Pend'g
9	Butt Welding Trials	Mon 6/2/03	Fri 6/6/03	Rev Pend'g
10	Attachment Welding Trials	Mon 6/2/03	Fri 6/6/03	Rev Pend'g
11	6A.2 Reporting	Fri 2/1/02	Fri 1/2/04	Rev Pend'g
12	6A.3 SH: Met Test & Eval	Mon 6/23/03	Wed 7/23/03	Rev Pend'g
13	6A.3.1 Mic Bends&Welds	Mon 6/23/03	Thu 8/14/03	Rev Pend'g
14	6A.3.2 HT Studies&Mic	Mon 6/23/03	Fri 9/5/03	Rev Pend'g
15	**6B: Fab Thk Wall Comp**	Thu 1/2/03	Fri 12/31/04	Rev Pend'g
16	6B.1 Thick-Wall Fab Trial	Fri 1/2/04	Mon 6/14/04	Rev Pend'g
17	6B.2 Reporting	Mon 2/2/04	Fri 12/31/04	Rev Pend'g
18	6B.3 Thick-Wall Comp	Fri 1/2/04	Fri 8/13/04	Rev Pend'g
19	6B.3.1 Met Analysis	Tue 6/15/04	Fri 10/15/04	Rev Pend'g
20	6B.3.2 PWHT Studies&Mic	Thu 7/15/04	Mon 11/1/04	Rev Pend'g



Task 7 Coatings (Alstom)

- The major objectives for Task 7 Coatings are:
  - Review state-of-the-art of coating technology and identify development needs.
  - Develop coating manufacturing techniques, which can provide corrosion/erosion protection for components in USC boilers, cost effectively.
  - Establish manufacturing techniques for application of internal coatings for oxidation protection, cost effectively.
  - Provide coated samples for corrosion and oxidation testing in the laboratory and "in the field".

These objectives will be accomplished through execution of eight sub-tasks. Where activity on these sub-tasks occurred during the reporting period, it is described below.

Task 7B: Coating Feasibility (Internal Coatings)

- Objective: The primary objective of this subtask is to examine internal tube coating techniques for oxidation protection.

- Progress for the Quarter: Continued evaluation of diffusion coating system details. Additional discussion of results is contained in an appendix to this report.

- Concerns: No concerns at this time.

- Plans for the Next Quarter: Qualification of alternative Si-Cr pack cementation formulations. T-23, T-92 and S304H coupons will be used.

Task 7C: Coating Recommendations

- Objective: Provide an evaluation of scaleup potential and costs for internal tube coating systems.

- Progress for the Quarter: Evaluation of diffusion coating system details needed to establish successful ID coating process. Additional discussion of results is contained in an appendix to this report.

- Concerns: No concerns at this time.

- Plans for the Next Quarter: Continue evaluation of ID coatings in sections of S304H and possibly T-92. Coatings will include chromizing, Si-Cr, and Al-Cr coatings.

Task 7D: Laboratory Testing

- Objective: Evaluate corrosion/oxidation response of candidate coating systems.
- Progress for the Quarter: Steam oxidation testing of substrate and coated specimens was stated. Detailed results are presented in an appendix to this report
- Concerns: None
- Plans for the Next Quarter: Continue testing of coating systems.

Task 7E: Process Scaleup

- Objective: Perform coating process trials at an intermediate scale between laboratory and commercial size.
- Progress for the Quarter:

Part 1: B&W Effort

Weld clad coupons were provided to Foster Wheeler for fireside corrosion assessment testing per Task 4. Coupons were prepared for three materials: IN 52, IN 72, and IN 622. The approach was to machine these coupons from deposited weld metal. Steps were taken to ensure that sufficient material was deposited to minimize dilution effects and ensure representative deposit chemistry. These all-weld-metal coupons were machined from weld pad build-ups that were prepared by a subcontractor, Welding Services, Inc (WSI). Use of all-weld-metal coupons eliminated concerns about edge effects and other complications implied by the required specimen design. WSI was selected to perform these build-ups because they are a vendor who might reasonably be employed to provide weld-clad tubes for commercial service.

A vendor, ASB, has been requested to quote sample preparation using the HVOF and “cold spray” deposition processes. The HVOF process was selected as being most likely to be able to deliver the required coating characteristics; however, “cold spray” is being considered as a state-of-the art alternative. The cold spray process offers the advantage that the deposition process entails minimal heat input. Instead, coating powder particles are propelled toward the coated surface using high velocity gas flow. The energy of impact is sufficiently high that a bond is achieved, in a similar manner to that which is achieved in explosion welding. Four coating types are being considered. One coating is a 50Cr-50Ni. The others are variations on MCrAlY.

Additional project plans have been sketched out where other coating approaches will be investigated. Laser cladding and plasma transferred arc deposition methods are being considered. The details of these will be fleshed out in the near future.

Part 2: Alstom Effort

Planning and logistics for execution of the scale-up efforts for internal coating of tubing with pack cementation techniques continued. Sufficient T-92 and Super 304H tubing has been ordered for 15 test runs with each test run processing nine 5-ft long sections. An existing gas-fired processing furnace is being refurbished for use in the test program.

- Concerns: None

- Plans for the Next Quarter: Complete preparations necessary for testing operations to begin in September, 2003.

Task 7H: Specimens for Field Corrosion/Oxidation

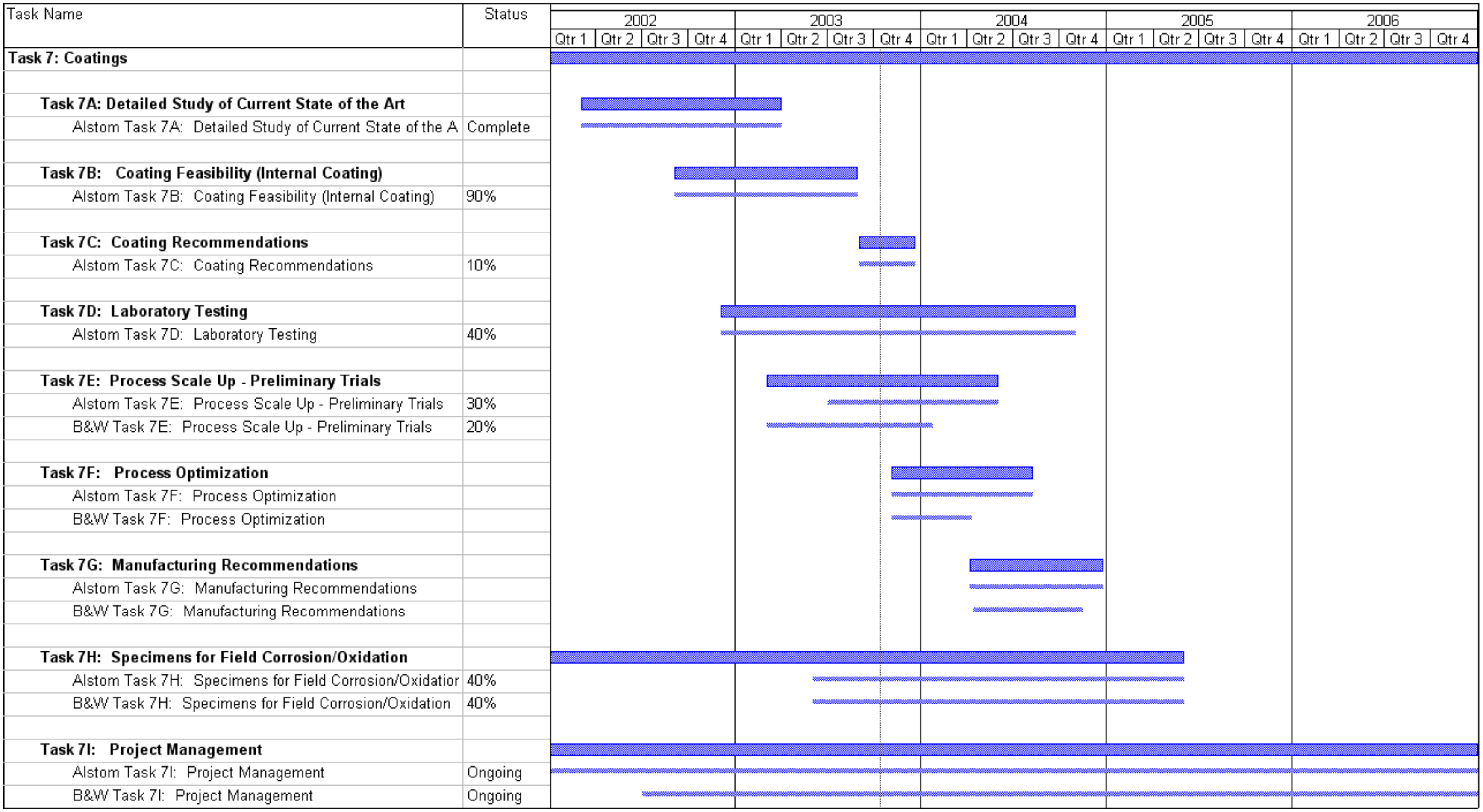
- Objective: Provide externally and internally coated specimens for inclusion in corrosion/oxidation testing under Tasks 3 and 4.

- Progress for the Quarter:
  - Super 304H specimens were diffusion coated with Cr, Al-Cr, and Si-Cr and supplied to Foster Wheeler for fireside corrosion testing under program Task 4.
  - Production of Super 304H coated tube specimens for the Niles steam loop was underway but not completed as of the end of the quarter.

- Concerns: None

- Plans for the Next Quarter:
  - Complete processing and deliver Super 304H coated tube samples to B&W for inclusion in the Niles steam test loop.
  - Continue processing of test samples to support Task 3 and 4 test schedules.

Task 7 Planning Chart
Task 7 Coatings



Task Name	Status
Task 7: Coatings	
Task 7A: Detailed Study of Current State of the Art	
Alstom Task 7A: Detailed Study of Current State of the A	Complete
Task 7B: Coating Feasibility (Internal Coating)	
Alstom Task 7B: Coating Feasibility (Internal Coating)	90%
Task 7C: Coating Recommendations	
Alstom Task 7C: Coating Recommendations	10%
Task 7D: Laboratory Testing	
Alstom Task 7D: Laboratory Testing	40%
Task 7E: Process Scale Up - Preliminary Trials	
Alstom Task 7E: Process Scale Up - Preliminary Trials	30%
B&W Task 7E: Process Scale Up - Preliminary Trials	20%
Task 7F: Process Optimization	
Alstom Task 7F: Process Optimization	
B&W Task 7F: Process Optimization	
Task 7G: Manufacturing Recommendations	
Alstom Task 7G: Manufacturing Recommendations	
B&W Task 7G: Manufacturing Recommendations	
Task 7H: Specimens for Field Corrosion/Oxidation	
Alstom Task 7H: Specimens for Field Corrosion/Oxidatior	40%
B&W Task 7H: Specimens for Field Corrosion/Oxidation	40%
Task 7I: Project Management	
Alstom Task 7I: Project Management	Ongoing
B&W Task 7I: Project Management	Ongoing

Note: Dates refers to DOE fiscal yar calendar 10/01/yyy to 09/03/yyy+1

Appendix to April-June 2003 Quarterly Report
Coatings Development

Introduction:

Coupon samples of T-92 and S304H were coated for B&W and Foster Wheeler to be included in their respective long-term laboratory tests. The diffusion coatings included straight chromizing, Si-Cr rich layers and Al-Cr rich layers. The T-92 samples will be tested in superheated steam and the S304H samples will be tested in coal-ash-corrosion test conditions. The characteristics of the coatings generated in control samples were documented and reported as part of the monthly reports for the month of April-2003. Additionally, samples of T-92 were also coated to support tests in superheated steam using a thermogravimetric Analytical balance (TGA). The results of these tests will provide an indication of the oxidation kinetics and of the benefits of diffusion coatings in steam oxidation.

Preliminary tests with tubing sections were initiated using a steel retort sealed with a low-melting glass. These tests have been conducted with T-23 tubing sections and most recently sections of S304H have been included. Particular attention has been paid to the characteristics of the ID diffusion layers. The results of these preliminary tests will be reported.

Additional experimental work has been conducted to define other formulations capable of generating acceptable Si-Cr containing layers. The new formulations will not be disclosed to avoid loosing intellectual property that may hamper any future economical benefits to the consortium. Preliminary results will be discussed regarding characteristics of the diffusion layers.

The infrastructure needed to support the scale-up trials is underway. T-92 and S304H tubing has been purchased together with the required consumables to conduct a minimum of 15 tests including straight chromizing, Si-Cr and Al-Cr containing diffusion layers. The materials were chosen based of the recommendations from Task 1. Nine (9) 5 ft-long tubing sections will be used per test in a 6 x 2 x 2 ft steel retort. Thermocouples will be installed strategically through the pack to monitor the heating and cooling cycles. Each load will generate coated tubing that can become available for other related post-tests including, post-heat treatments, welding and bending. These series of tests are scheduled to start in the month of September.

Discussion

Research activities continue evaluating alternative formulations to generate a Si-Cr rich diffusion layer. This effort is driven by the difficulties experienced in sourcing CaF_2 of the desired specifications in bulk quantities. The approaches include using both different activators and inert fillers. Figure 1depicts the Si-concentration profiles obtained in T-23 tubing sections using two formulation variants and they are compared with the profile from a diffusion layer generated using the CaF_2-base formulation. Si-Sand correspond to a recipe using chloride-base activators and sand as the filler material. The Si-content across the diffusion layer is half the concentration of Si obtained using the qualified process with CaF_2 as the activator. A modification to the activator system will be implemented shortly. This change, based on thermodynamic calculations, may generate a layer of adequate characteristics. Results should become available in the July monthly report.

The process designated as Thermi, was designed based on the instability of silica in the presence of metallic aluminum. Even though silicon is introduced in the diffusion layer, the concentration across the layer is about one-third the concentration obtained using the qualified process. This particular approach requires tuning of the formulation and a modification to the constituent ratio is planned within the next series of tests. The reduction in the diffusion layer thickness is noticeable with both the Si-Sand and the Si-Thermi approach, about 200 μm thinner than the layer obtained with the CaF2 activator. A reduction in the Cr-content across the diffusion layer is also apparent, Figure 2.

The coating process in the ID of tubing components using the pack cementation approach is limited by the throw power generated within the tube which in turn is related to the total amount of powder available in the ID for the coating process. T-23 tubing sections 3 inch-long and with an ID of 0.75 inch were used to conduct these tests. Starting with the formulation in US Patent 5,972,429, both the Si and Cr concentrations in the pack were increased. Each tubing section was packed with 50 g of powder. Figure 3 shows the concentration profiles for Si in the diffusion layers generated in T-23 test pieces. A gradual increase in the silicon content from 2 to 3 w% resulted in a concentration profile similar to that obtained in the OD using the qualified formulation. An appreciable change in the Cr-concentration was not apparent as indicated by Figure 4. The diffusion layer was continuous with no apparent intergranular porosity. The contour of the ID coating was somehow roughing, typical of the Si-Cr layers obtained using this formulation.

The quality of ID chromized layers was also improved by changing the source of Cr in the straight chromizing pack. Pure chromium was used instead of Fe-Cr for the ID coating process. The amount of chromium was gradually increased from 25 to 30 wt%. Figure 5 shows the Cr-concentration profiles. The larger concentration across the layer is obtained using the pack with 30 wt% Cr. The coating profile was smooth with no apparent intergranular porosity.

Tests were initiated with S304H type of material and the preliminary results will be reported in the month of July. The ID of this tubing is larger and therefore we do not expect any significant variation between OD and ID formulations. In addition to chromizing and Si-Cr, tests will be conducted with the Al-Cr formulation.

The characteristics of the aluminized layers in T-92 were determined in samples prepared for the TGA tests. Figure 6 depicts the diffusion layer. There is an outward growing layer about 30 μm thick with an average Al and Cr content of 8 and 16 wt%, respectively. This outward growing layer is not continuous since it tends to fell off during handling due to the weak bonding to the inner growing layer. Fine alumina particles are usually found at the interface between both layers and this causes the outer layer to spall off. The coating layer also shows an array of precipitates that are Al-nitride.

Figure 7 shows the average concentration profiles for Al and Cr across the layer. The coating thickness is about 700 μm. As a reminder, this layer was generated using the qualified formulation by Rapp but extending the processing time to 16 h. Processing periods shorter than 16 h are unrealistic from the point of view of the infrastructure required to heat up large loads of tubing. An increase processing time has resulted in a lower nominal concentration for Al across the layer. Our target was 10 wt% or higher maintaining at least the same Cr-content of the base alloy. Processing times of 6 hours delivered layers with Al-contents between 10 and 14 wt%. There was significant scattered in the concentration profiles by as much as ± 2 wt%. The longer process time apparently reduces the scattered generating layers that are more consistent in thickness and composition. Even though the maximum Al concentration is less than the desirable value of 10 wt%, the diffusion layer seems to provide adequate protection as indicated by short term TGA tests at 650 C. Figure 8 compares the Al concentration profiles before and after an oxidation test in superheated steam at 650 C. There is no apparent change in the composition of the diffusion layer after exposure. Aluminide coatings generally change in time upon exposures to temperatures in excess of half the melting point of Al. Aluminum continues diffusing into the alloy base and the Al concentration and coating/metal interface are continuously changing due to a simultaneous oxidation and inter-diffusion processes. This sample did not experience oxidation attack and the results in Figure 8 suggest that at least for short exposure times, the diffusion layer is stable. More detail will be provided in another section of the overall quarterly report for Task 7. Similarly, the chromized samples of T-92 had shown no degradation after 160 hour tests in superheated steam at 650, 700 and 750 C. As expected, these diffusion layers significantly improve the oxidation resistance of the bare T-92 material.

Task 7E Scale-up trials are scheduled to begin in September 2003. S304H and T-92 tubing have been purchased to conduct a minimum of 15 tests. Nine (9) 5-ft long sections will be used per test and thermocouples will be place inside the retort to monitor the heating and cooling characteristics of each pack. A steel retort 2x2x6 ft will be used with the lid welded. A gas-fired furnace is being refurbished to conduct these tests. Post-test analysis will include destructive metallographic evaluation of the center tube and of the ends of the corner tubes. In a pack cementation process of this scale, the center tube is the one that normally gets to temperature and cools down the last. For ferritic alloys cooling rates are critical in relation to carbide precipitation. Therefore, it is considered that the center tube may provide the worst possible scenario generating a more a realistic expectation on product quality. The corner tubes may represent the best possible product. Completion of this subtask is expected by May 2004.

Future work:

Task 7b:

Task 7c:

Task 7d:

Evaluation of coated samples provided to B&W and Foster Wheeler. Progress in this task will depend on the particular testing schedules.

Task 7e:

Finalize the acquisition of consumables to support the test matrix.

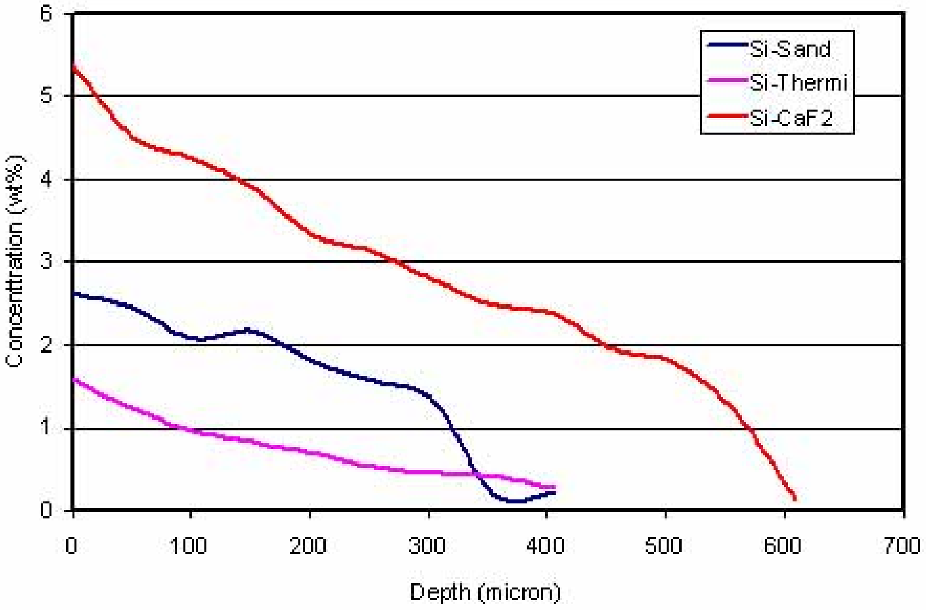


Figure 1. Comparison of Si-content profiles generated in the Si-Cr diffusion layers in T-23 using various formulations.

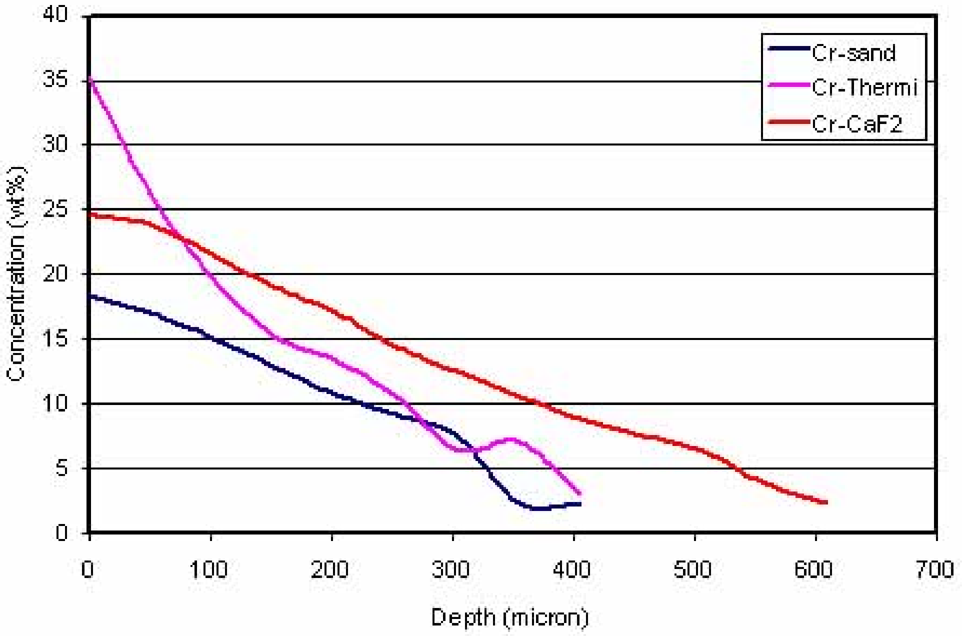


Figure 2. Comparison of Cr-content profiles generated in the Si-Cr diffusion layers in T-23 using various formulations.

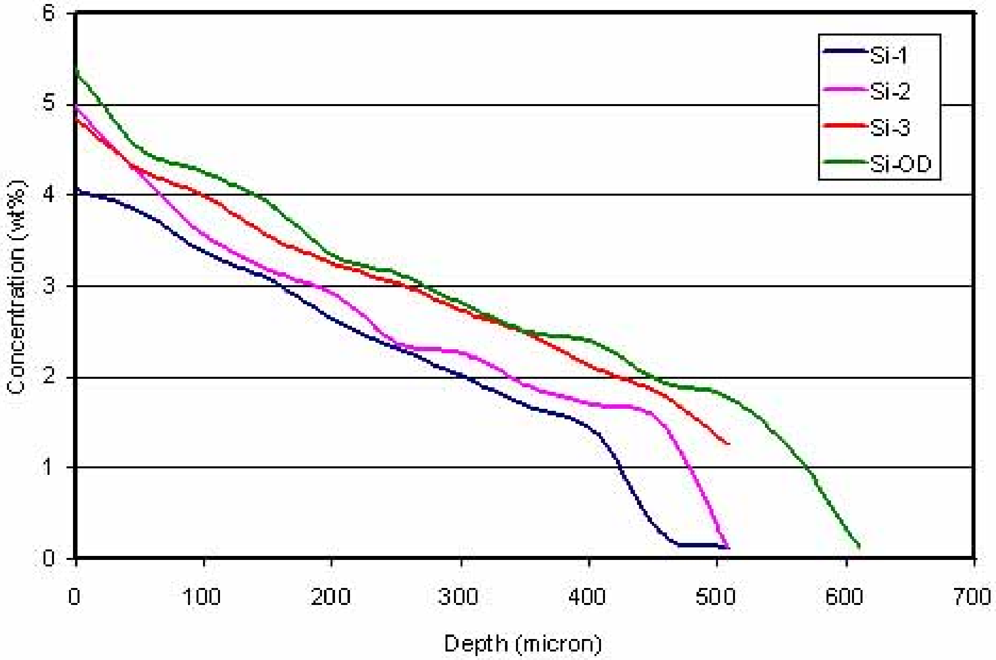


Figure 3. Comparison of Si-content profiles in the ID and OD of T-23 tubing pieces.

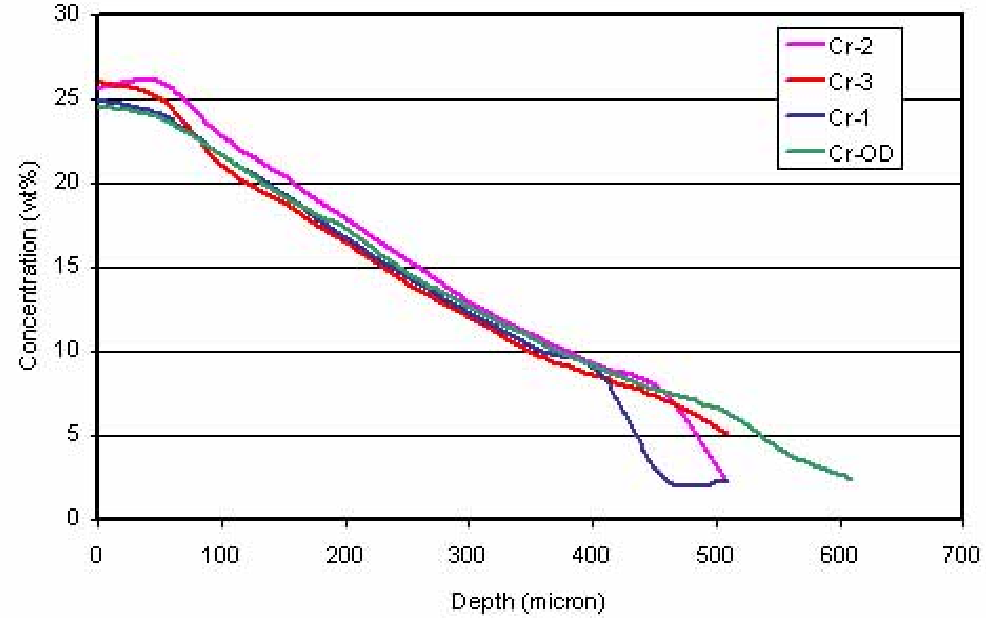


Figure 4. Comparison of Cr-content in the ID diffusion layers generated in pieces of T-23 tubing.

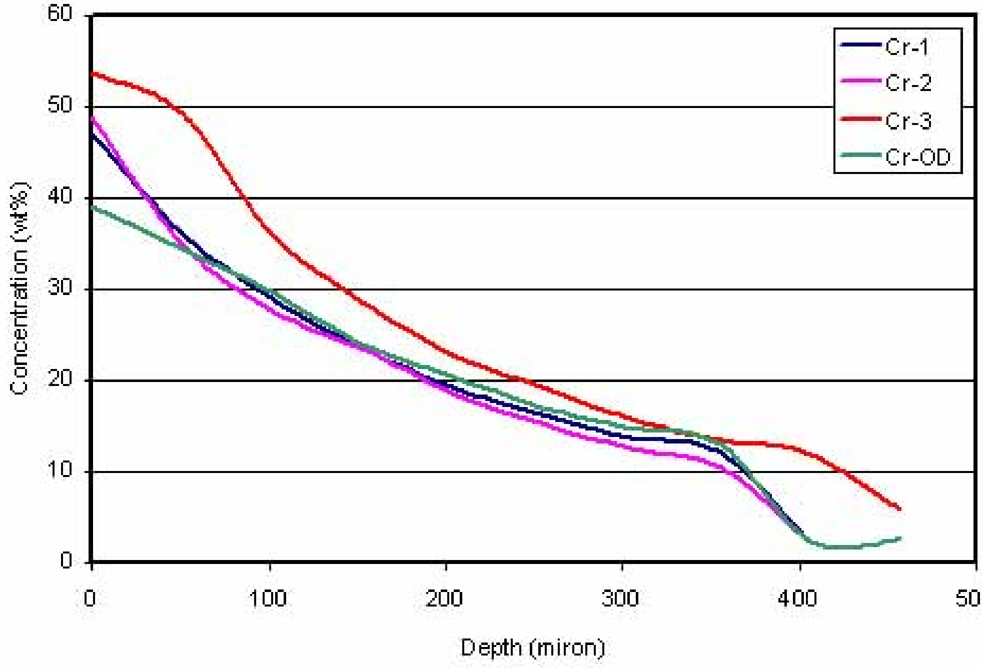


Figure 5. Comparison of Cr-content in the ID and OD of chromized pieces of T-23 tubing.

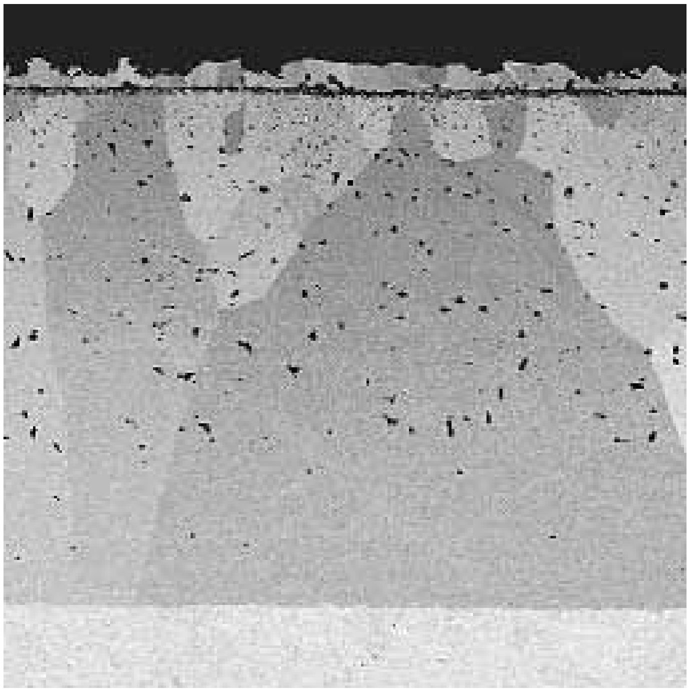


Figure 6. Backscattered Image from the Al-Cr diffusion coating generated in T-92 type of material.

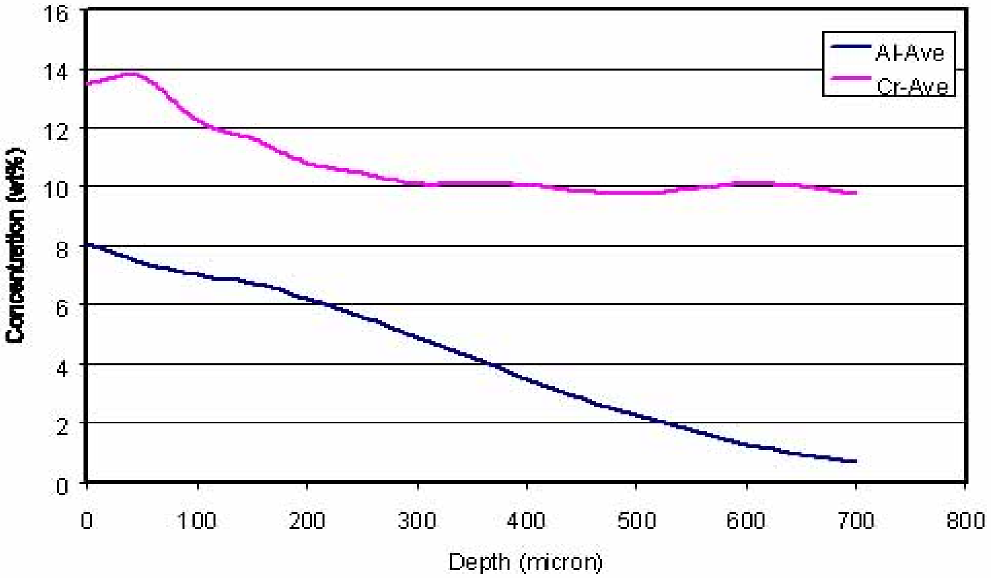


Figure 7. Average Al and Cr concentration profiles through diffusion layer generated in T-92 material.

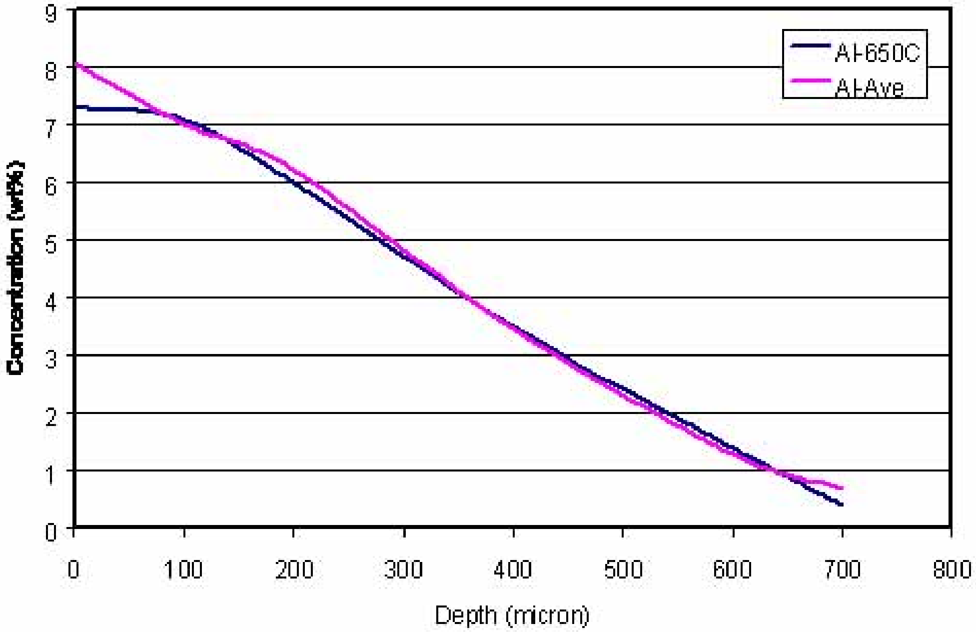


Figure 8. Comparison of Al-Concentration profile through the Cr-Al diffusion layer after exposure to superheated steam at 650 C for 162 h.

Appendix to April-June 2003 Quarterly Report
Evaluation of Steam Oxidation Testing

Introduction

Short-term steam oxidation tests by thermogravimetric analysis (TGA) were conducted on various samples of Grade 22, Grade 92 and Super 304H material to evaluate coating performance and oxidation resistance at 650, 700 and 750°C. Following post-test weight gain determinations, samples were examined in cross-section by optical microscopy to characterize the coating or oxidized surface, and by SEM/EDS to determine compositional profiles and the extent of oxidation. Unless otherwise indicated, scanning electron micrographs were acquired in the backscattered electron mode. The current report summarizes the findings of the tests performed in this reporting period.

Results

The samples tested and examined for this reporting period include:
1) Grade 22 high-phosphorous electroless nickel (650, 750°C)
2) Grade 92 uncoated (650°C)
3) Grade 92 with Cerablak coating (650°C)
4) Grade 92 chromized (650, 700 and 750°C)
5) Grade 92 with Si-Cr layer (650°C)
6) Grade 92 with Al-Cr layer (650°C)
7) Super 304H (650°C)

Weight Gain

Weight gain results as measured by TGA are presented in Table 1 for an exposure time of approximately 150 h. It is seen that four of the five coated samples tested at 650°C experienced minimal weight gains (<0.4 mg/cm^2). The highest weight gains were observed in the uncoated Grade 92 sample (11.1 mg/cm^2) and the Grade 92 Cerablak coated sample (8.8 mg/cm^2) tested at 650°C. Super 304 H experienced minimal weight gain (0.1 mg/cm^2) at 650°C. Chromized Grade 92 samples showed similar, low weight gain responses for 650, 700 and 750°C exposures. The weight gain for nickel-coated Grade 22 tested at 750°C was about an order of magnitude higher than that tested at 650°C.

Metallography and SEM/EDS

1) Grade 22 high-phosphorous electroless nickel (650°C, 750°C)

Optical and scanning electron micrographs depicting the nickel coated surface for the 650°C sample are shown in Figures 1 and 2, respectively. SEM images were acquired using the backscattered electron mode to better distinguish surface layers. EDS analysis results (Table 2) suggest that the thin (1μm) outermost film (Area 1) consists of iron and nickel oxides. This film appears to be detached from the surface. Below this film is a 16 μm layer (Area 2) composed of the Ni-P coating. Below this layer is a 12 μm layer (Area 3) consisting of an Fe-Ni intermetallic phase with traces of Grade 22 alloy additions. In the optical micrograph, the intermetallic appears as a white-colored layer. The composition of Area 4 reflects the base metal constituents. Overall, exposure at 650°C revealed excellent short-term oxidation performance. However, the diffusion of nickel into the substrate is indicative of instability in the coating structure, which may be problematic for long service periods.

In contrast, the sample tested at 750°C exhibited considerable oxidation as illustrated in Figures 3 and 4. An 18 μm layer consisting of iron and nickel oxide covered the surface. The upper portion of the oxide was buckled (Figure 4, Area 1). Below this oxide is a 15 μm layer (Area 3) composed of the Ni-P coating. Below this layer is a 9 μm zone of oxidation containing iron oxide within the Ni-P layer. This zone marks the point where the Ni-P coating is becoming detached from the 12 μm Fe-Ni intermetallic phase (Area 5). Below the Fe-Ni phase is a 40 μm thick zone of internal oxidation (Area 6). The composition of Area 4 reflects the base metal constituents.

These observations are consistent with the weight gain differences between the 650°C and 750°C samples. Recall that the weight gain was an order of magnitude greater in the 750°C sample. None the less, the short-term oxidation protection offered at a temperature far above normal service usage temperature for this alloy is quite remarkable.

2) Grade 92 uncoated

A cross-sectional view of the Grade 92 surface is shown in Figure 5, with corresponding SEM images of the same area shown in Figures 6 and 7. The oxide is seen to consist of two layers of equal thickness (60 µm). EDS results show that the inner layer (Area 5) consists of an Fe, Cr spinel containing Grade 92 alloying constituents. The outer layer (Area 4) consists of magnetite. The boundary between these two layers is the original tube surface. The magnetite layer itself appears to be delaminating. The optical micrograph (Figure 5) shows a thin band (about 4-5 µm) of a lighter colored oxide at the surface (Area 2 in Figure 7) and a thin (2 µm) layer of exfoliated oxide flakes above the surface (Area 1 in Figure 7), suggestive of hematite.

3) Grade 92 with Cerablak Coating

Optical and scanning electron micrographs of the Cerablak-coated Grade 92 surface are shown in Figures 8 and 9, respectively. The two-layer structure of the oxide is clearly depicted, with each layer being approximately 35 µm thick. EDS results indicate the inner layer to consist of an Fe, Cr spinel while the outer layer is magnetite. The outermost 10 µm of the magnetite layer has delaminated. There is no evidence of the Cerablak coating. The coating vendor, Applied Thin Films, has suggested that a pre-exposure anneal in air may be beneficial to coating performance. This will be done in a future trial.

4) Grade 92 Chromized (650°C, 700°C, 750°C)

The optical micrograph and SEM image of the chromized surface on the 650°C sample (Figures 10 and 11, respectively) illustrate the subsurface porosity (Kirkendal effect) that is limited to within about 30 µm from the surface. The chromized layer is approximately 350 µm thick. The concentration profile (Figure 12) showed a maximum chromium concentration of 26.3 wt. % at a depth of 10 µm and a minimum concentration of 12 wt% at the base metal interface.

Samples tested at 700°C (Figures 13-15) and 750°C (Figures 16-19) also showed subsurface porosity extending about 40 µm from the surface. The chromized layer thickness for these samples was 450-500 µm. Concentration profiles for the 700°C sample (Figure 15) showed a maximum chromium concentration of 51.4 wt. % at the surface and a minimum concentration of 10.5 wt% at the base metal interface. For the 750°C sample (Figure 19), the maximum chromium concentration was 29.8 wt. % at a depth of 30 µm and a minimum concentration of 10.0 wt% at the base metal interface.

The SEM image (Figure 18) was taken in the secondary electron mode to illustrate a very thin film at the surface (Area 1) which EDS results indicate to be iron and chromium rich oxide. The film is <1µm thick. The light-colored rim (Area 2) around the void as well as the other similarly colored areas were found to consist predominantly of chromium.

5) Grade 92 Si-Cr

Optical and scanning electron micrographs of the Grade 92 Si-Cr surface are shown in Figures 20 and 21, respectively. The irregularly shaped protrusions that covered most of the surface are inherent to the coating, as they also appear in as-received samples. The Si-Cr layer was about 570 µm thick. The concentration profile (Figure 22) shows the highest chromium (~ 25wt.%) and silicon (~ 2 wt%) levels at 1-30 µm depths. The lowest chromium levels were about 10% near the base metal interface. Silicon was not detected near the base metal interface.

6) Grade 92 Al-Cr

The post-test characteristics of the Al-Cr diffusion coating are illustrated in the optical micrograph (Figure 23) and SEM images (Figures 24 and 25). The Al-Cr layer was about 800 μm thick. The micrographs clearly depict a 400 μm thick zone containing aluminum nitride precipitates and voids resulting from aluminum nitride dropout. Above this zone, at the surface (Area 1), the coating is discontinuous with a high void density. At a depth of about 30 μm from the original surface is a 12 μm thick continuous band (Area 2) that EDS results show to contain iron, chromium, aluminum and some oxygen. This band is also observed in as-received samples and has been found to contain alumina powder from the pack. The concentration profile (Figure 26) shows the highest chromium (~14 wt%) and aluminum (~7 wt%) levels in the upper 50 μm of the layer. The post-test characteristics of the coating are identical to those in as-received samples.

6) Super 304H

The optical micrograph (Figure 27) shows a representative view of the surface. There is no apparent oxide layer following exposure at 650°C. The SEM images (Figures 28, 29) illustrate some superficial (<1 grain deep) and localized oxidation. There were only a few of these penetrations over the entire surface of the coupon. The EDS results suggest oxide formation within the penetration and in the area above the surface. The penetration also contained the typical alloying additions, however, the surface oxide was composed of only Fe, Cr and Si in addition to the oxygen present. These isolated oxide penetrations are not uncommon in stainless steels.

Conclusions

- Chromium-based diffusion coatings (Cr, Al-Cr, Si-Cr) on Grade 92 material provided excellent short-term steam oxidation resistance in 650°C test environments. Chromized Grade 92 tested at 700°C and 750°C also showed excellent oxidation resistance.

- The high phosphorous electroless nickel coating on Grade 22 material provided excellent short-term steam oxidation resistance in the 650°C test environment. When tested at 750°C, however, the coating was significantly degraded, resulting in partial detachment of the Ni-P alloy from the underlying Fe-Ni intermetallic phase, and internal oxidation below the intermetallic. The weight gain (oxidation rate) was an order of magnitude greater at the higher test temperature.

- Super 304H exhibited excellent steam oxidation resistance in the 650°C environment but was subject to highly localized superficial surface penetrations.

    - Uncoated and Cerablak-coated Grade 92 samples experienced the highest weight gains in the steam oxidation tests. Classic duplex oxides were formed in the 650 °C steam environment. Total oxide thickness ranged from about 70 μm (Cerablak coated) to 120 μm (uncoated). Delamination of the outer magnetite layer was observed along with exfoliation of thin surface oxide films.

Table 1

Summary of Weight Gain Results from Steam Oxidation Tests

Sample	Test Temperature (°C)	Wt. Gain (mg/cm^2)
Grade 22 high-P electroless Ni	650	0.4
Grade 22 high-P electroless Ni	750	4.9
Grade 92 uncoated	650	11.1
Grade 92 w/Cerablak coating	650	8.8
Grade 92 chromized	650	0.3
Grade 92 chromized	700	0.2
Grade 92 chromized	750	0.2
Grade 92 with Si-Cr layer	650	0.1
Grade 92 with Al-Cr layer	650	0.1
Super 304 H	650	0.1

Table 2

Summary of EDS Analysis Results (Wt. %)

Sample	Fe	Cr	Ni	Mn	O	Si	Mo	W	Other
Grade 22 EN 650C									
Area 1	53.7	0.4	27.0	0.3	16.9	1.3	---	---	0.3 P
Area 2	1.6	---	84.3	---	---	---	---	---	14.1 P
Area 3	44.3	1.2	53.2	0.3	---	0.3	0.7	---	---
Area 4	93.9	3.5	---	0.8	---	0.4	1.7	---	---
Grade 22 EN 750C									
Area 1	78.3	0.4	4.9	0.7	15.5	0.2	---	---	---
Area 2	75.9	0.3	9.3	---	14.1	0.4	---	---	---
Area 3	10.5	---	75.9	---	---	---	---	---	13.6 P
Area 4	51.4	2.7	10.5	3.3	16.7	1.0	---	---	14.4 P
Area 5	54.7	1.5	42.9	0.3	---	0.2	0.5	---	---
Area 6	75.3	5.8	---	0.9	13.2	2.3	2.3	---	0.3 P
Area 7	96.3	2.2	---	0.5	---	0.2	0.7	---	---
Grade 92 650C									
Area 1	80.7	---	---	0.5	17.9	0.3	---	---	0.6 Cu
Area 2	80.7	---	---	0.6	18.2	0.4	---	---	---
Area 3	83.5	---	---	0.6	15.7	0.1	---	---	---
Area 4	83.2	---	---	0.6	16.2	---	---	---	---
Area 5	58.3	18.9	---	---	16.1	---	1.2	5.1	0.4 V
Grade 92 Cerablak 650C									
Area 1	82.2	0.2	---	---	17.4	0.2	---	---	---
Area 2	82.8	0.2	---	1.0	15.7	0.4	---	---	---
Area 3	83.6	0.3	---	---	15.9	0.2	---	---	---
Area 4	62.3	17.1	---	---	14.3	---	1.0	4.8	0.5 V
Area 5	58.6	20.2	---	---	13.8	---	1.2	5.8	0.4 V
Grade 92 Cr 650C									
1 μm depth	72.0	24.5		---			0.5	3.1	
10 μm depth	70.2	26.3		---			0.4	3.1	
20 μm depth	70.4	25.6		---			0.7	3.3	
30 μm depth	71.5	25.1		---			0.3	3.1	
100 μm depth	74.9	21.8		---			0.3	2.9	
150 μm depth	78.1	19.1		---			0.5	2.4	
200 μm depth	79.9	16.3		0.8			0.7	2.3	
250 μm depth	81.5	15.1		0.5			0.3	2.7	
300 μm depth	83.6	13.4		0.4			0.3	2.3	
350 μm depth	84.5	12.0		0.6			0.4	2.5	

Table 2 (cont.)

Summary of EDS Analysis Results (Wt. %)

Sample	Fe	Cr	Ni	Mn	O	Si	Mo	W	Other
Grade 92 Cr 700C									
1 µm depth	40.5	51.4		---			1.6	6.6	
10 µm depth	64.0	32.2		---			0.5	3.3	
20 µm depth	64.3	32.0		---			0.5	3.2	
30 µm depth	65.0	31.4		---			0.3	3.3	
50 µm depth	66.4	30.0		---			0.5	3.1	
100 µm depth	71.4	25.6		---			0.1	2.9	
150 µm depth	76.4	21.1		---			0.2	2.3	
200 µm depth	79.0	18.2		---			0.4	2.4	
250 µm depth	81.1	15.8		---			0.5	2.6	
300 µm depth	83.2	14.9		---			0.6	2.5	
350 µm depth	84.9	12.8		---			0.3	2.1	
400 µm depth	85.4	11.7		0.8			0.2	2.0	
450 µm depth	85.5	11.1		0.6			0.6	2.2	
500 µm depth	85.7	10.5		0.8			0.5	2.5	
Grade 92 Cr 750C									
1 µm depth	70.1	27.3		---	---	---	---	2.6	---
10 µm depth	68.0	29.1		---	---	---	---	2.9	---
20 µm depth	67.9	29.1		---	---	---	---	3.0	---
30 µm depth	66.7	29.8		---	---	---	0.6	2.9	---
50 µm depth	67.6	29.5		---	---	---	---	2.9	---
100 µm depth	72.1	24.9		---	---	---	0.4	2.6	---
150 µm depth	76.1	20.9		---	---	---	0.2	2.7	---
200 µm depth	79.3	18.0		---	---	---	0.5	2.2	---
250 µm depth	81.4	15.5		---	---	---	0.5	2.6	---
300 µm depth	82.6	13.8		0.5	---	---	0.5	2.6	---
350 µm depth	83.9	12.3		0.8	---	---	0.5	2.5	---
400 µm depth	85.1	11.5		0.6	---	---	0.3	2.0	0.3 V
450 µm depth	86.5	10.3		0.6	---	---	0.5	2.1	---
Area 1 (thin film)	86.4	10.0		0.5	---	---	0.3	2.5	---
Area 2 (white rim)	46.0	43.5		---	8.3	1.3	---	---	0.8 V
	17.2	73.2		---	---	---	2.2	7.4	---

Table 2 (cont.)

Summary of EDS Analysis Results (Wt. %)

Sample	Fe	Cr	Si	Al	O	Mo	W	Other
Grade 92 Si-Cr								
1 µm depth	71.9	25.9	2.2			---	---	
10 µm depth	72.1	25.8	2.0			---	---	
20 µm depth	72.0	25.4	2.1			0.6	---	
30 µm depth	72.7	24.7	2.3			0.2	---	
100 µm depth	77.9	19.8	1.9			0.5	---	
150 µm depth	80.9	17.2	1.6			0.4	---	
200 µm depth	80.9	15.8	0.9			0.5	2.0	
250 µm depth	82.6	14.0	0.7			0.7	2.0	
300 µm depth	83.5	13.3	0.4			0.5	2.3	
400 µm depth	85.7	11.4	0.5			0.4	2.0	
500 µm depth	87.0	10.4	---			0.3	2.3	
570 µm depth	86.1	10.2	---			0.4	2.5	
Grade 92 Al-Cr								
1 µm depth	76.6	14.7		7.3	---	---	1.4	---
10 µm depth	75.7	14.7		7.6	---	0.4	1.6	---
20 µm depth	76.2	14.3		7.6	---	0.2	1.7	---
30 µm depth	76.6	14.1		7.5	---	0.3	1.4	---
50 µm depth	77.2	13.5		7.4	---	0.4	1.5	---
100 µm depth	78.8	12.5		7.1	---	---	1.6	---
200 µm depth	80.7	10.8		6.0	---	0.3	2.3	---
400 µm depth	83.2	10.1		3.5	---	0.6	1.8	0.8 Mn
600 µm depth	85.5	9.6		1.4	---	---	2.9	0.5 Mn
720 µm depth	88.3	8.5		0.4	---	---	2.1	0.6 Mn
Area 1 (surface)	68.7	16.5		7.5	---2.5	0.1	1.7	---
Area 2 (interface)	69.9	14.6		11.7	---	---	1.4	---
Area 3 (precipitate)	5.5	0.9		77.4		0.9	---	14.7 N
Super 304H								
Area 1 (surface oxide)	63.5	19.8	0.9	---	15.9	---		---
Area 2 (interior)	31.0	38.6	0.6	0.2	13.1	0.3		10.4 Ni, 4.2 Cu, 0.4 Nb, 1.1 Mn
Area 3 (base metal)	67.8	20.0	0.2	---	---	0.3		8.6 Ni, 2.6 Cu, 0.4 Nb

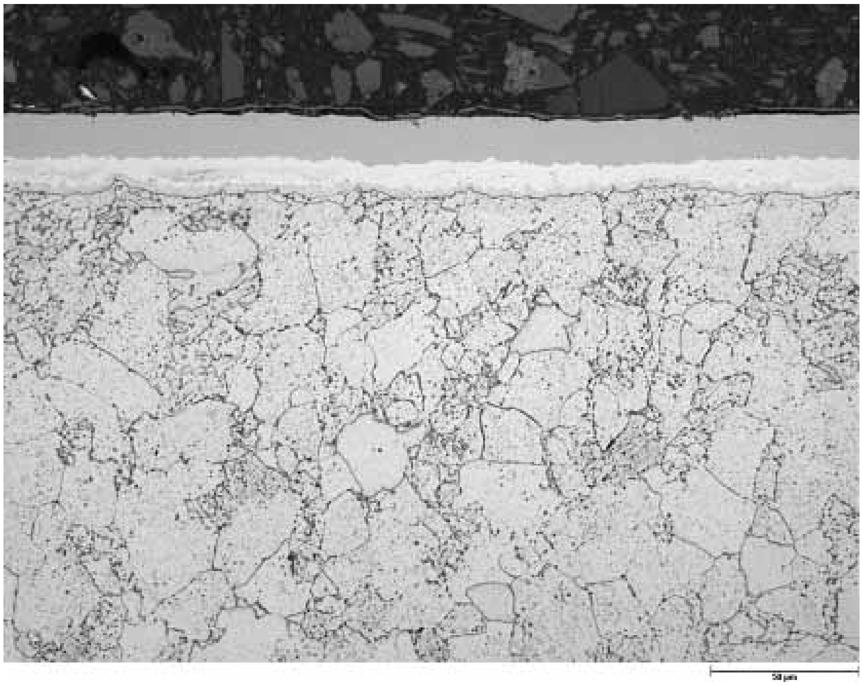


Figure 1: Optical Micrograph of Grade 22 Hi-P Ni Sample (650°C)

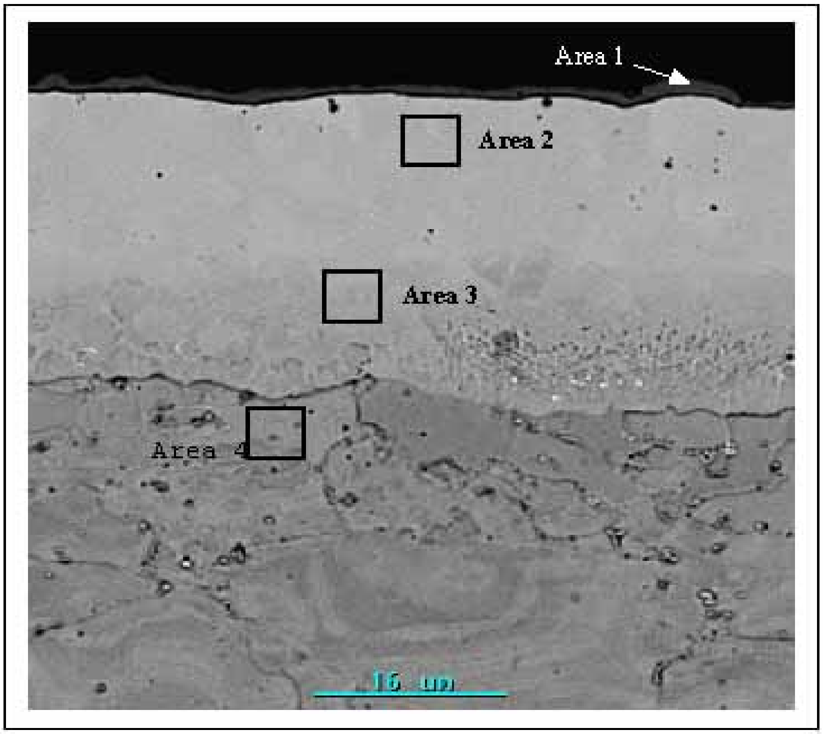


Figure 2: SEM Image of the Surface on the Grade 22 Sample (650°C)

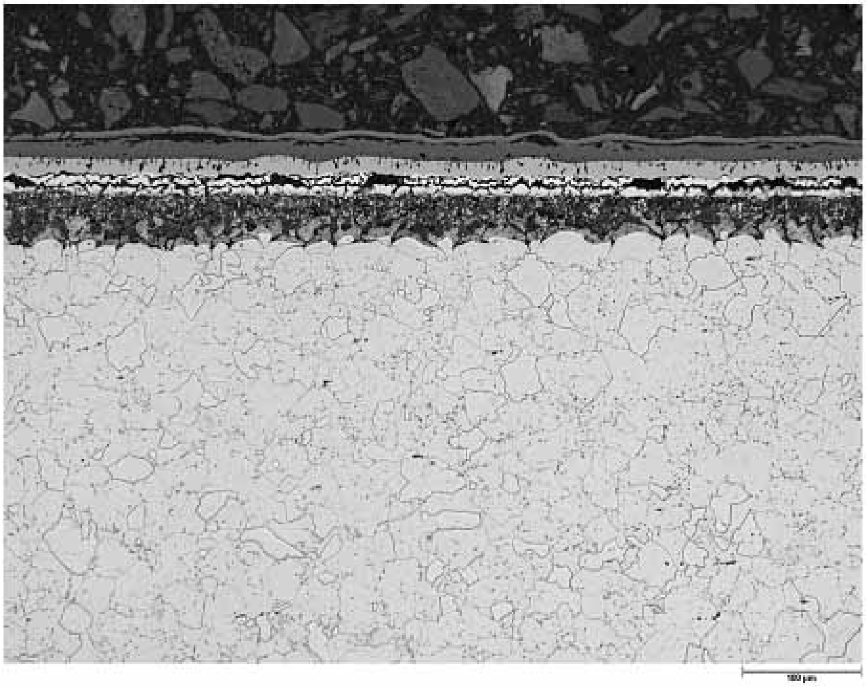


Figure 3: Optical Micrograph of Grade 22 Hi-P Ni Sample (750°C)

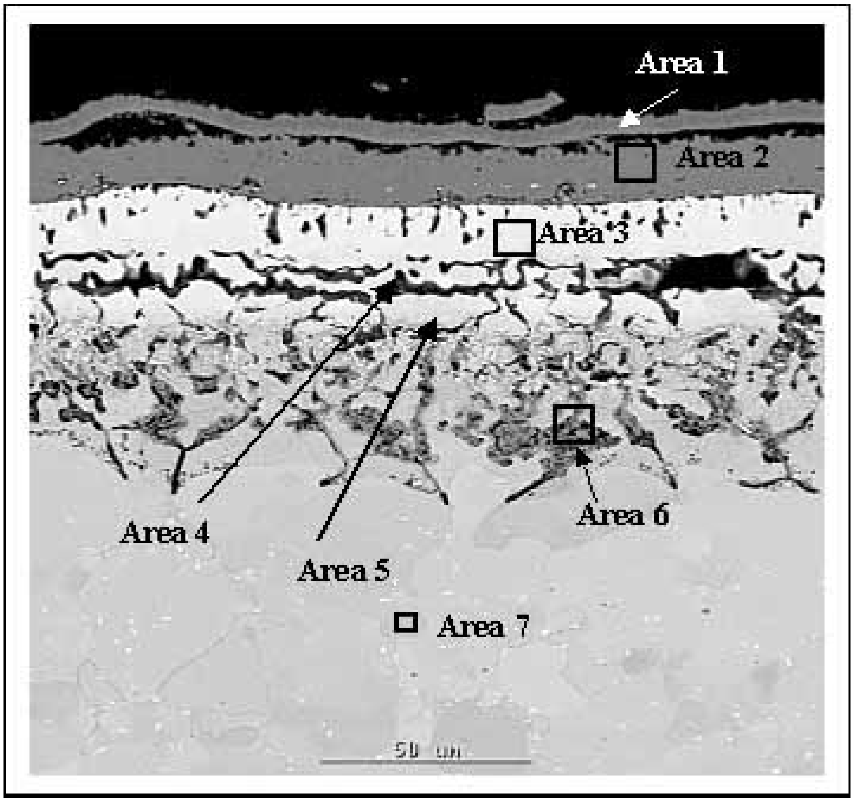


Figure 4: SEM Image of the Surface on the Grade 22 Sample (750°C)

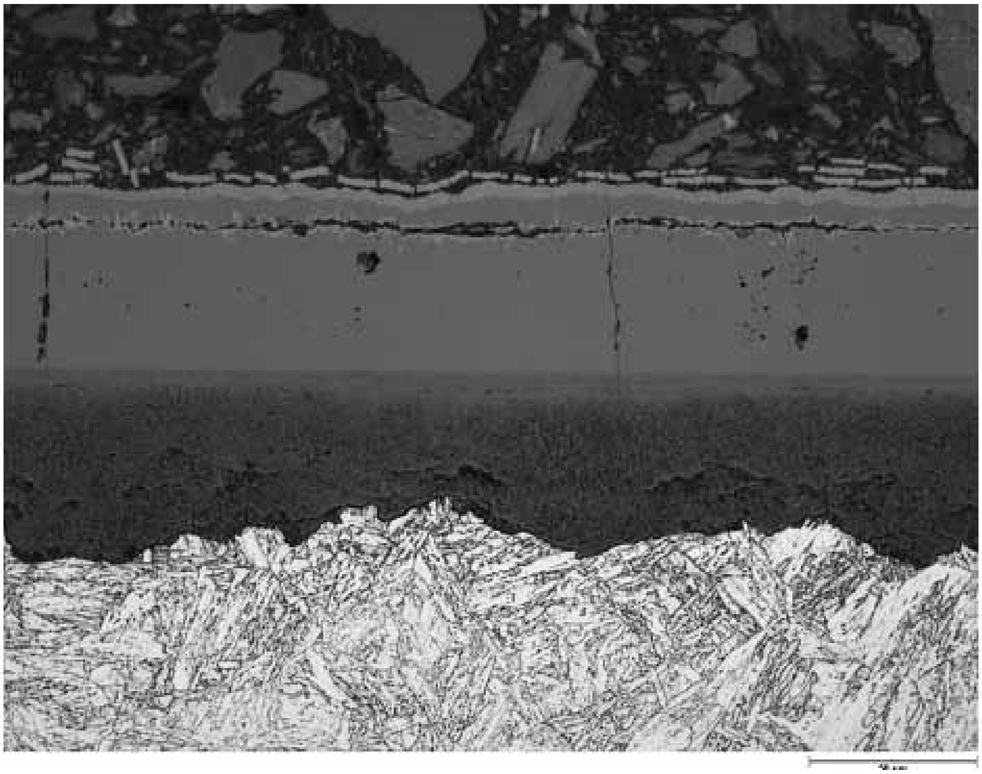


Figure 5: Optical Micrograph of the Uncoated Grade 92 Sample (650°C)

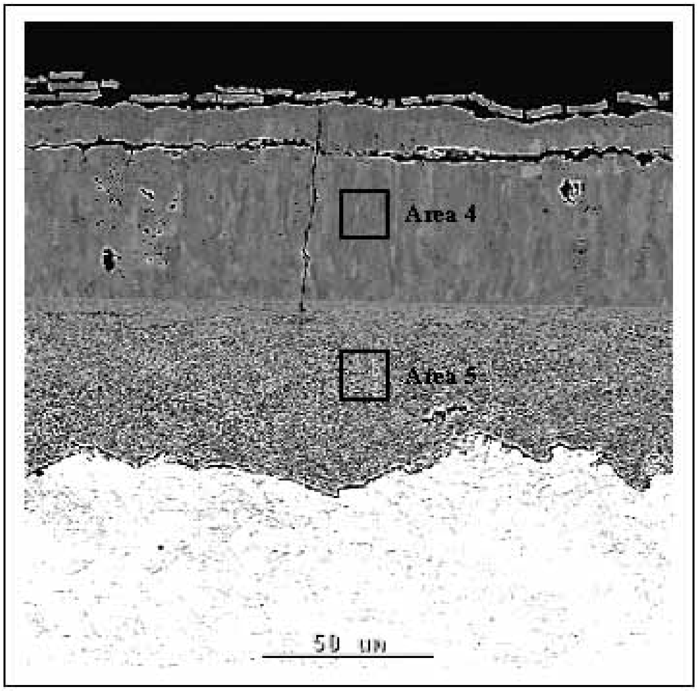


Figure 6: SEM Image of the Uncoated Grade 92 Surface (650°C)

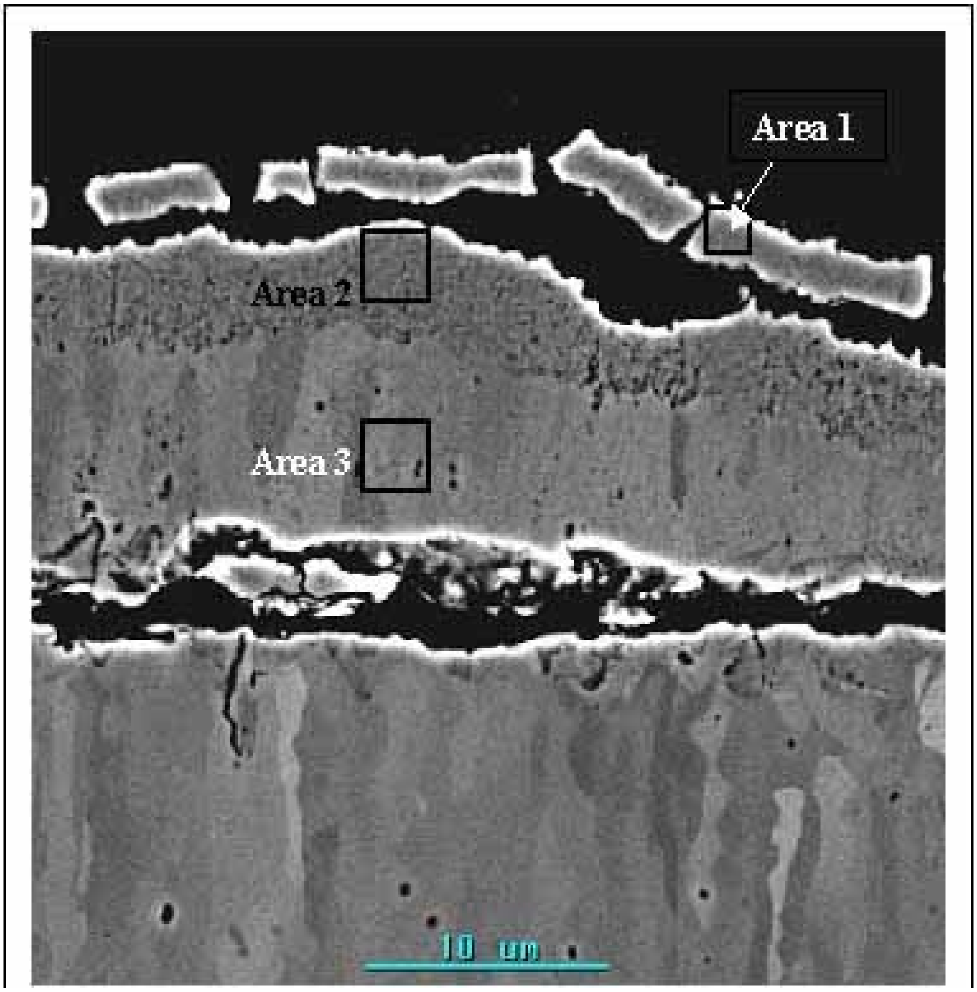


Figure 7: Higher Magnification SEM Image of the Uncoated Grade 92 Surface (650°C)

Figure 8: Optical Micrograph of the Grade 92 Cerablak Coated Sample (650°C)

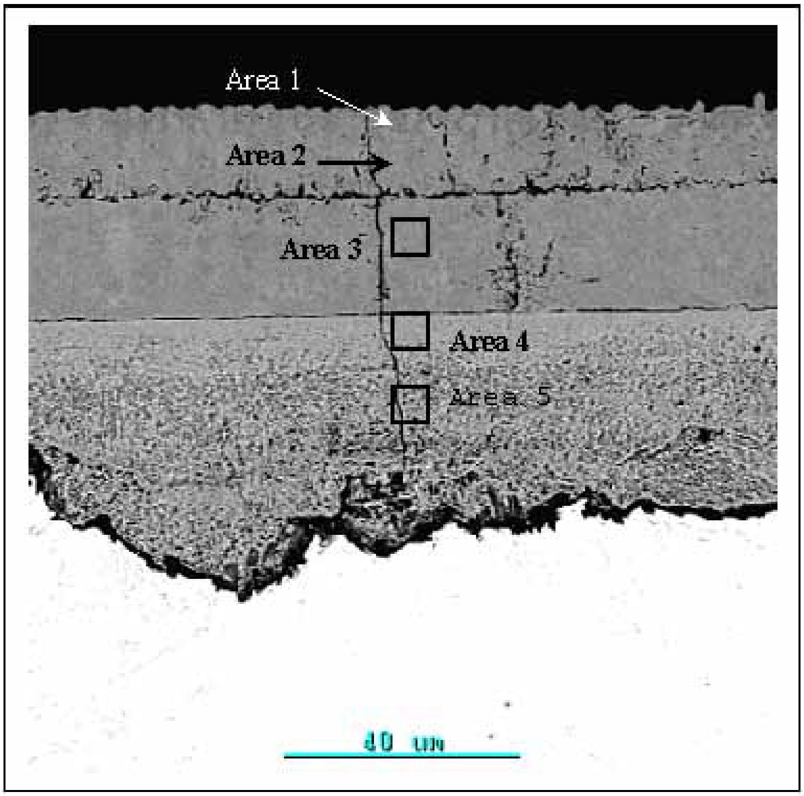


Figure 9: SEM Image of the Grade 92 Cerablak Coated Surface (650°C)

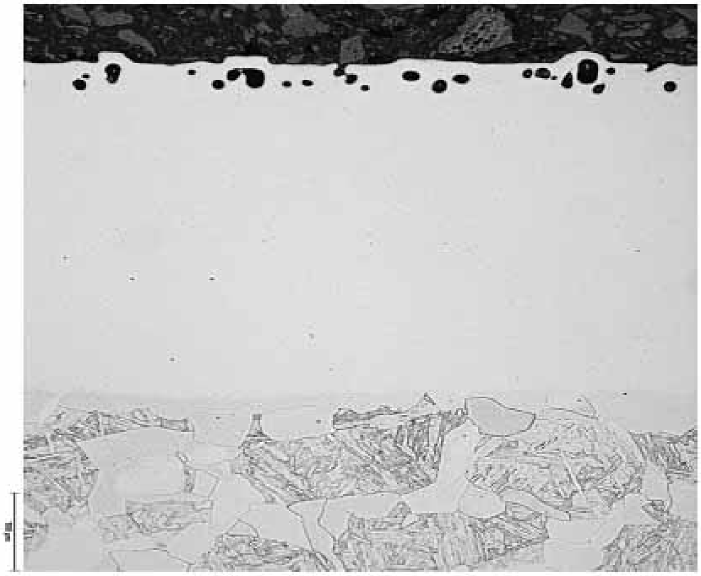


Figure 10: Optical Micrograph of the Grade 92 Chromized Sample (650°C)

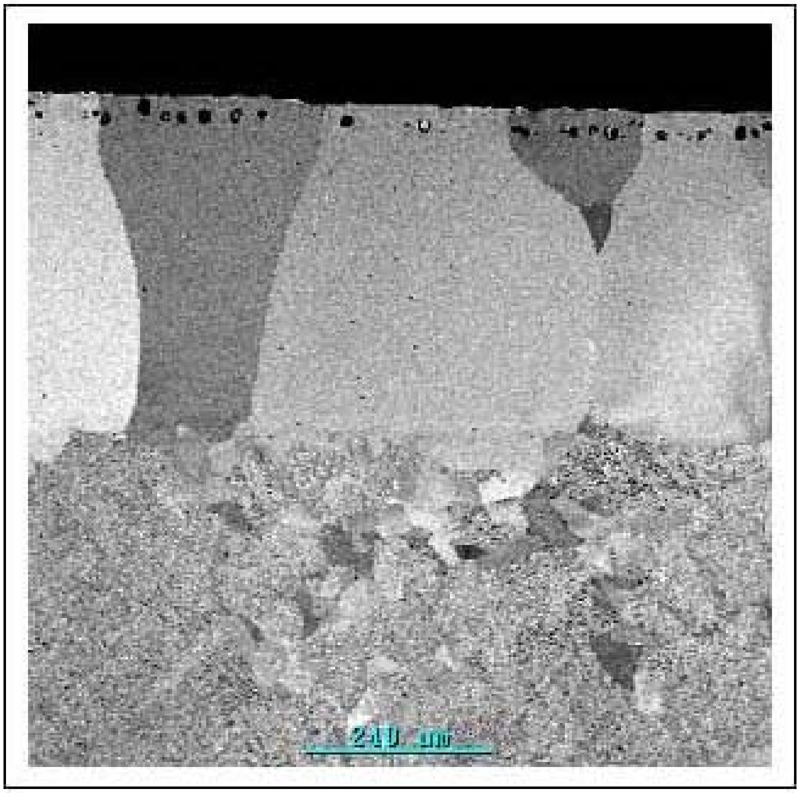


Figure 11: SEM Image of the Grade 92 Chromized Surface (650°C)

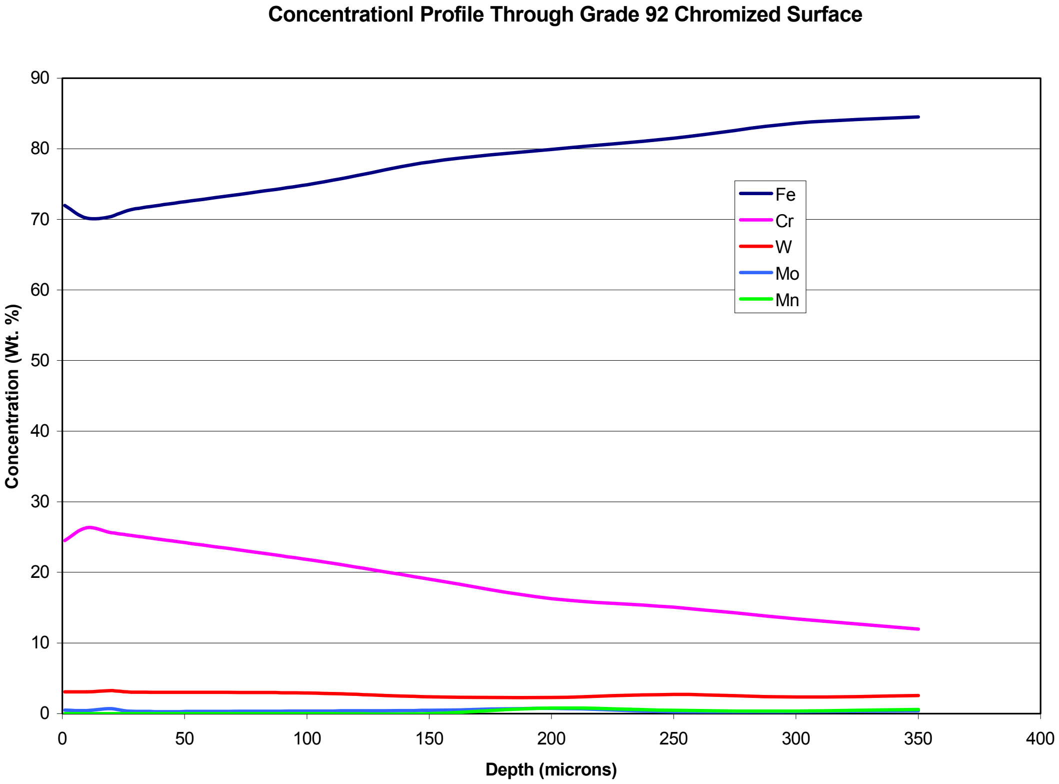


Figure 12: Concentration Profile for Chromized Layer on Grade 92 (650°C)

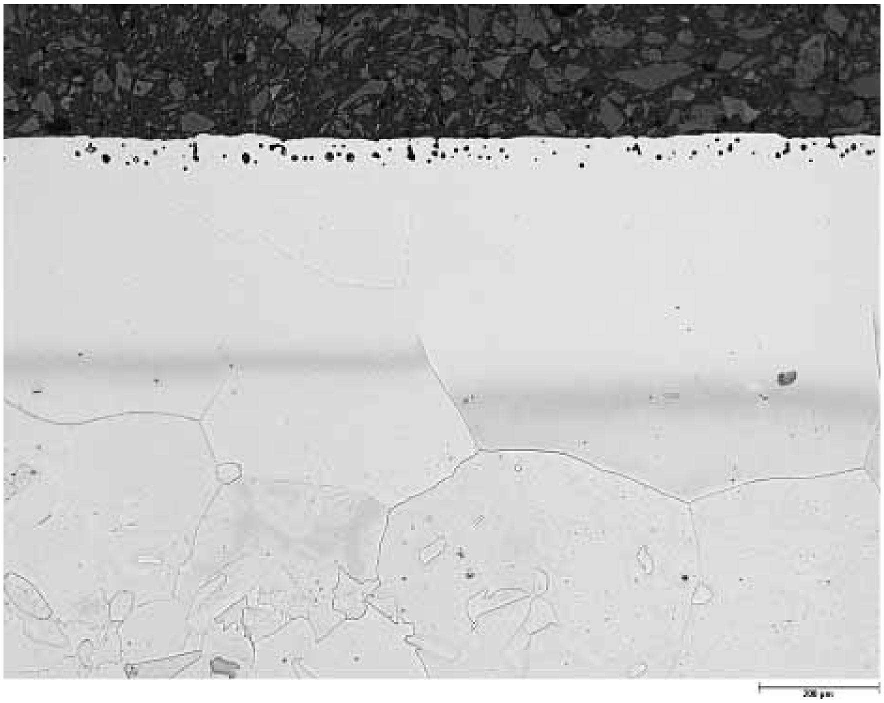


Figure 13: Optical Micrograph of the Grade 92 Chromized Sample (700°C)

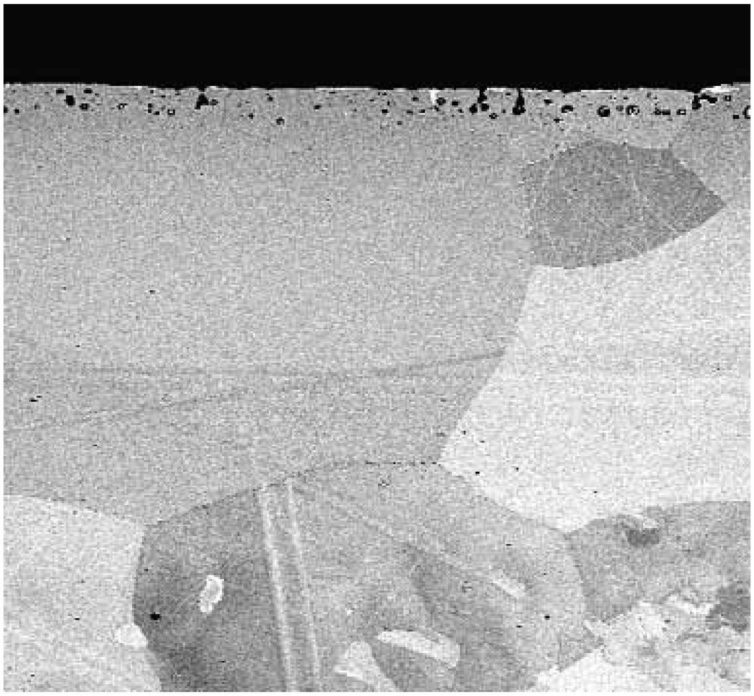


Figure 14: SEM Image of the Grade 92 Chromized Surface (700°C)

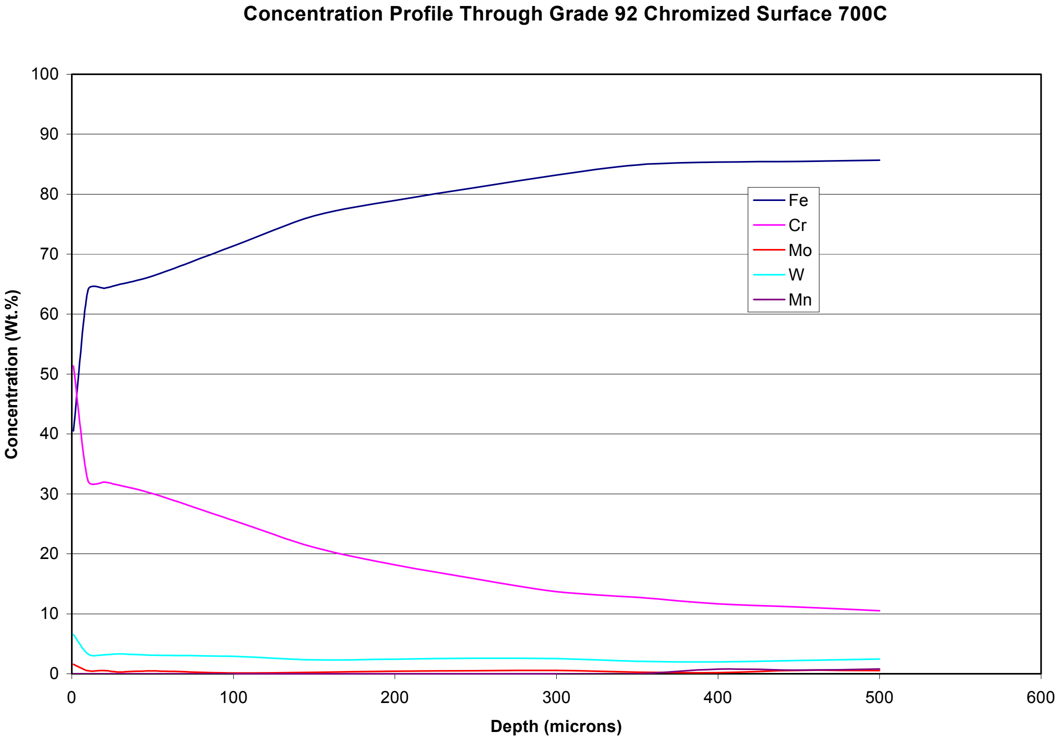


Figure 15: Concentration Profile for Chromized Layer on Grade 92 (700^{o}C)

Figure 16: Optical Micrograph of the Grade 92 Chromized Sample (750°C)

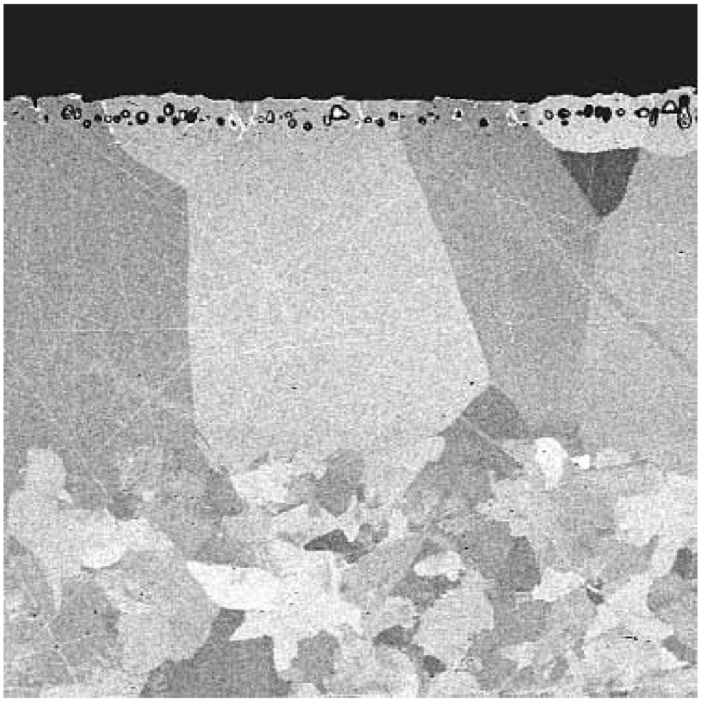


Figure 17: SEM Image of the Grade 92 Chromized Surface (750°C)

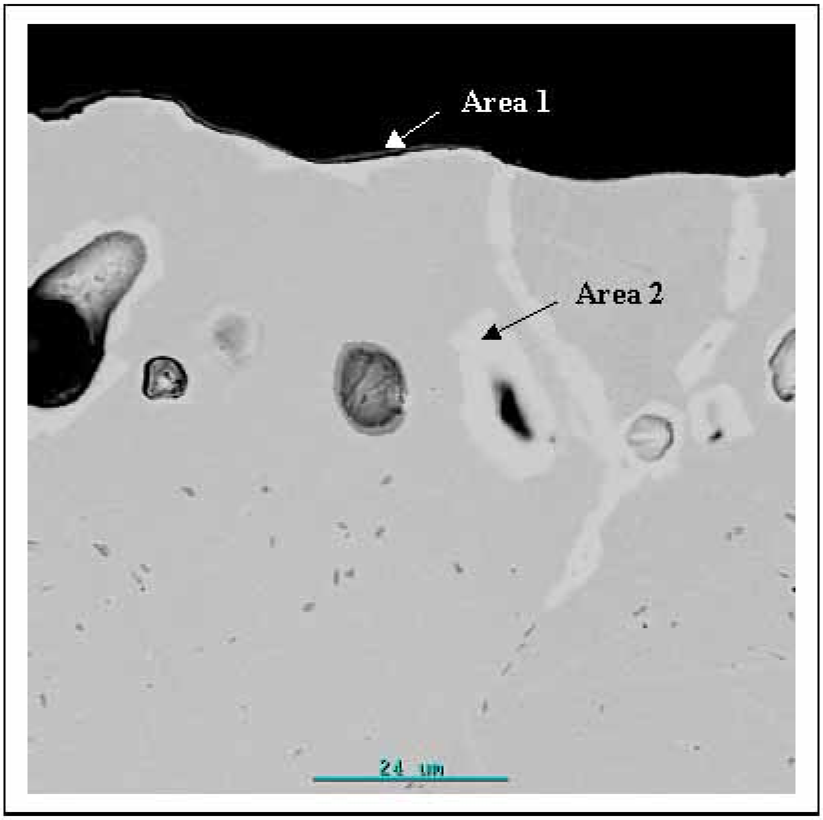


Figure 18: Secondary Electron SEM Image of the Grade 92 Chromized Surface (750°C)

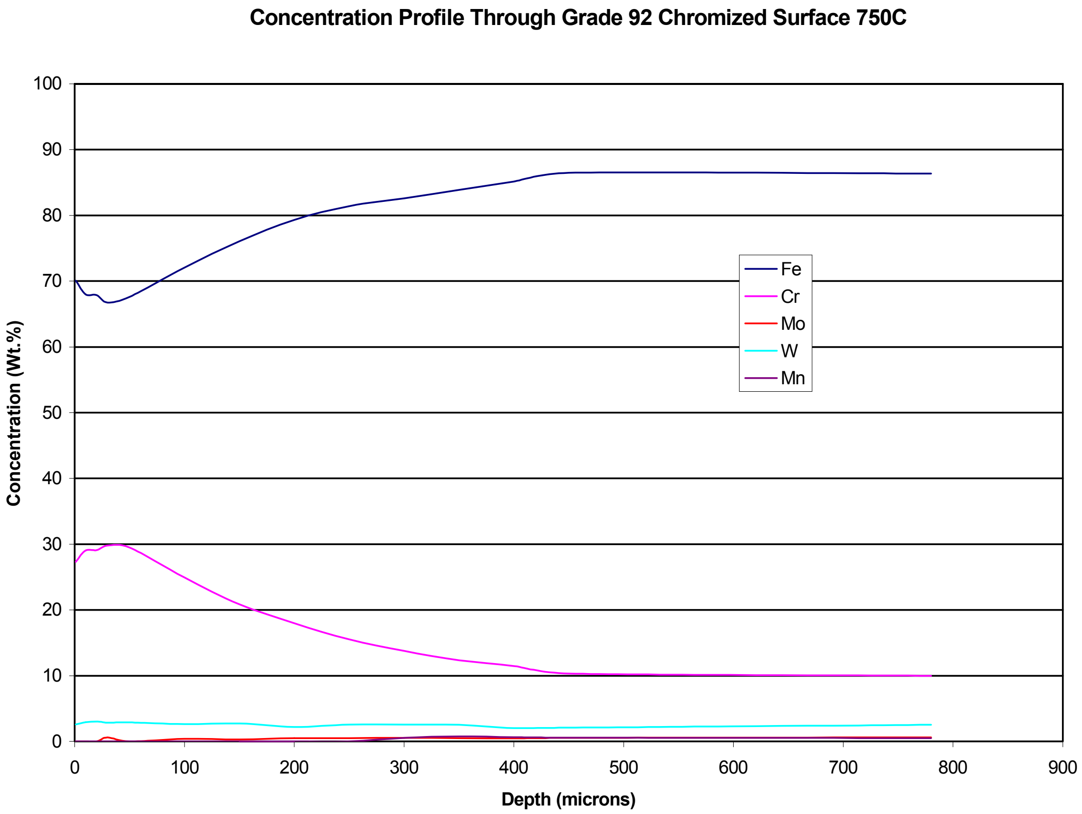


Figure 19: Concentration Profile for Chromized Layer on Grade 92 (750^{o}C)

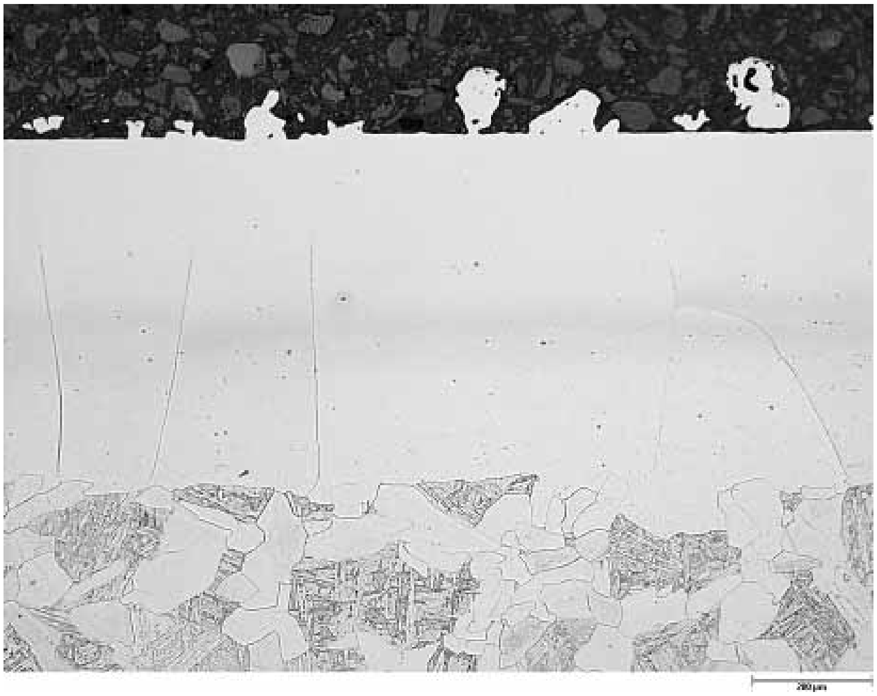


Figure 20: Optical Micrograph of the Grade 92 Si-Cr Sample (650°C)

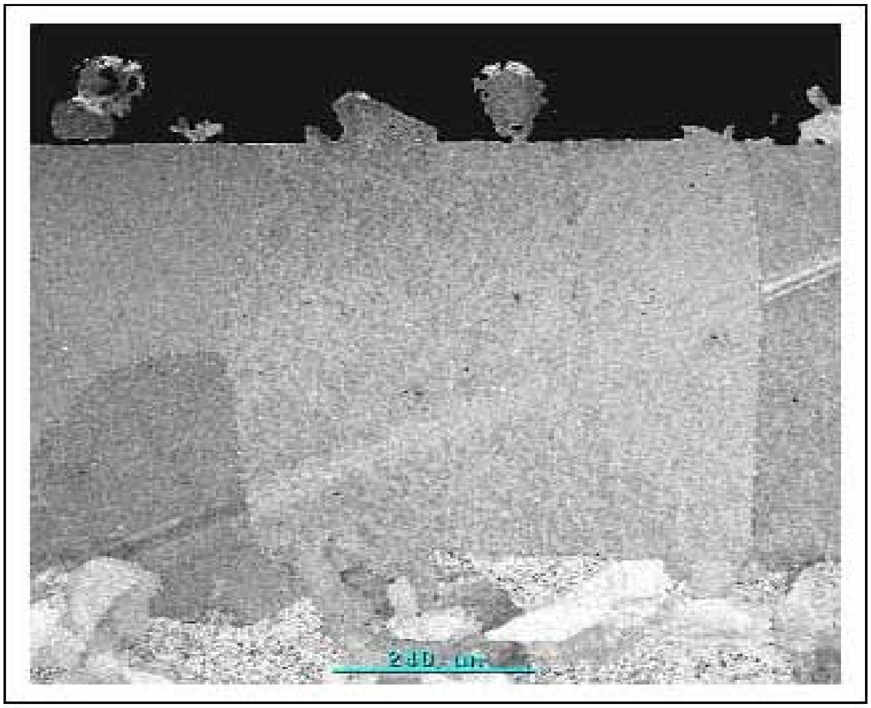


Figure 21: SEM Image of the Grade 92 Si-Cr Surface (650°C)

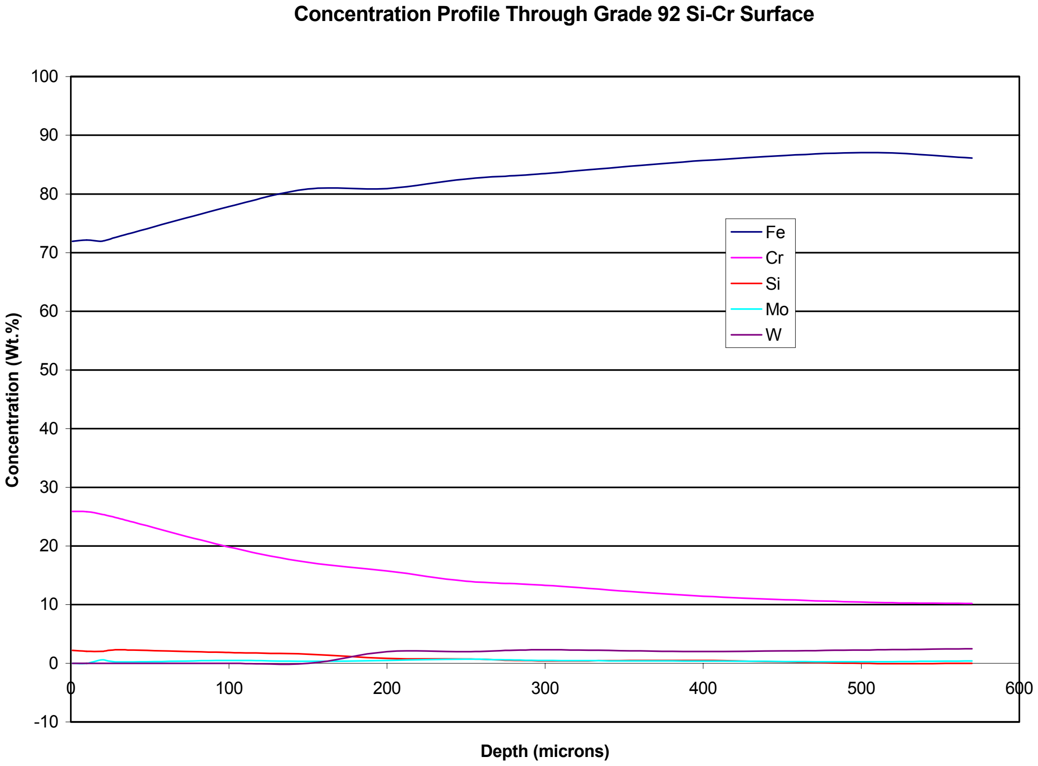


Figure 22: Concentration Profile for Si-Cr Layer on Grade 92 (950^{o}C)

Figure 23: Optical Micrograph of the Grade 92 Al-Cr Sample (650°C)

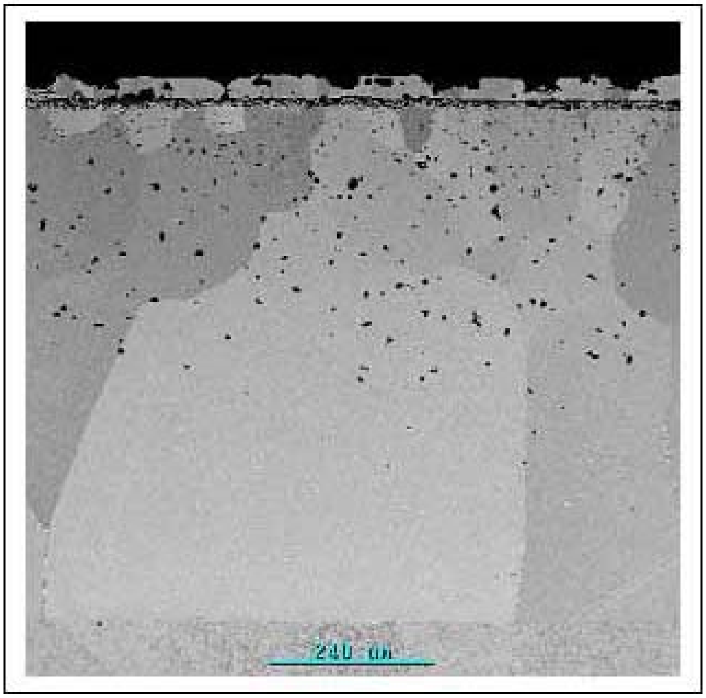


Figure 24: SEM Image of the Grade 92 Al-Cr Surface (650°C)

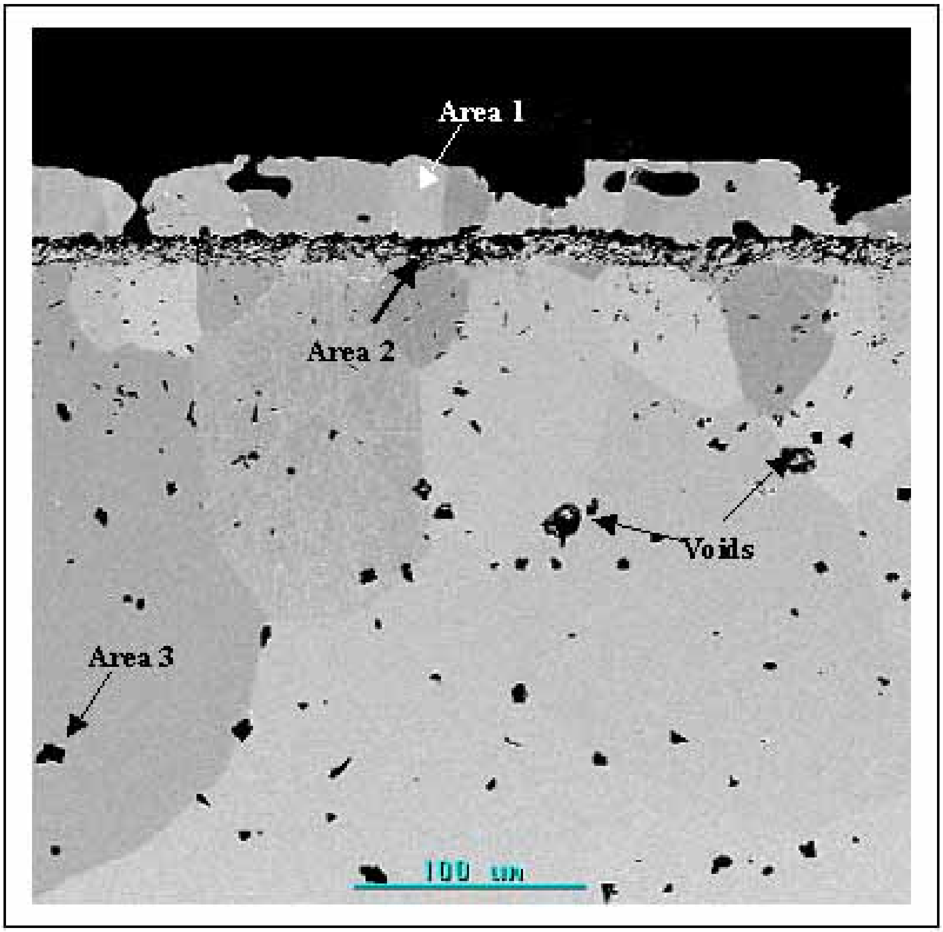


Figure 25: Higher Magnification SEM Image of the Grade 92 Al-Cr Surface (650°C)

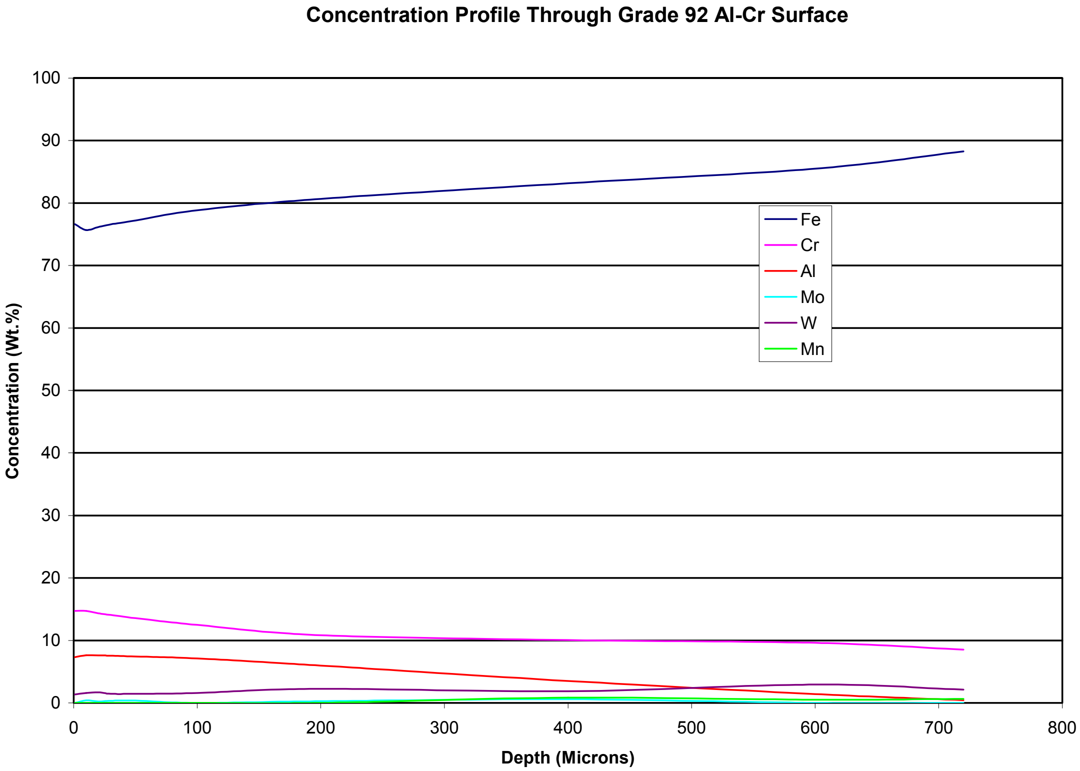


Figure 26: Concentration Profile for Al-Cr Layer on Grade 92 (650°C)

Figure 27: Optical Micrograph of the Super 304H Sample (650°C)

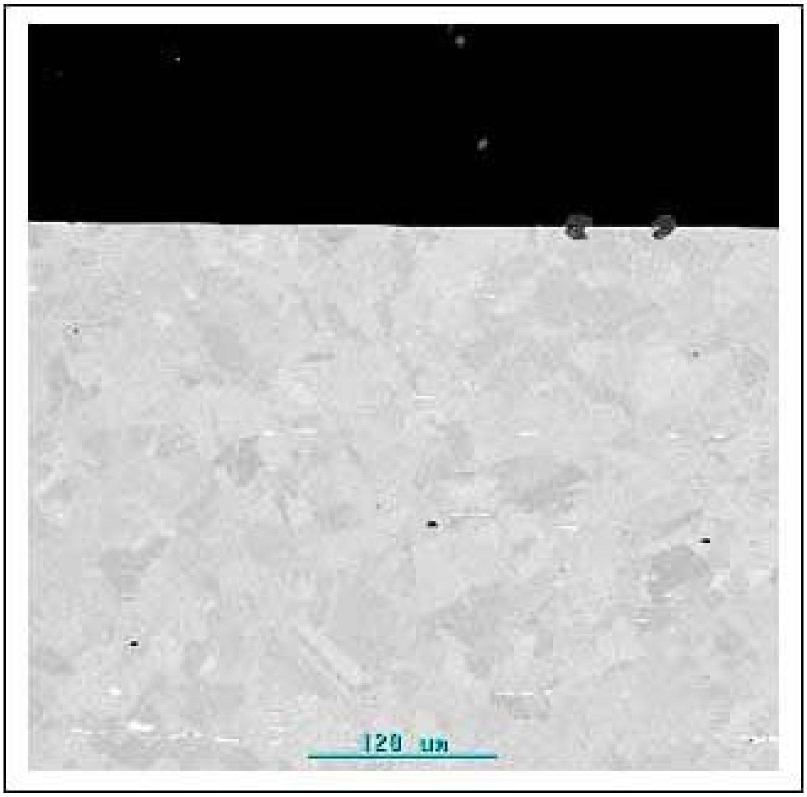


Figure 28: SEM Image of the Super 304H Surface (650°C)

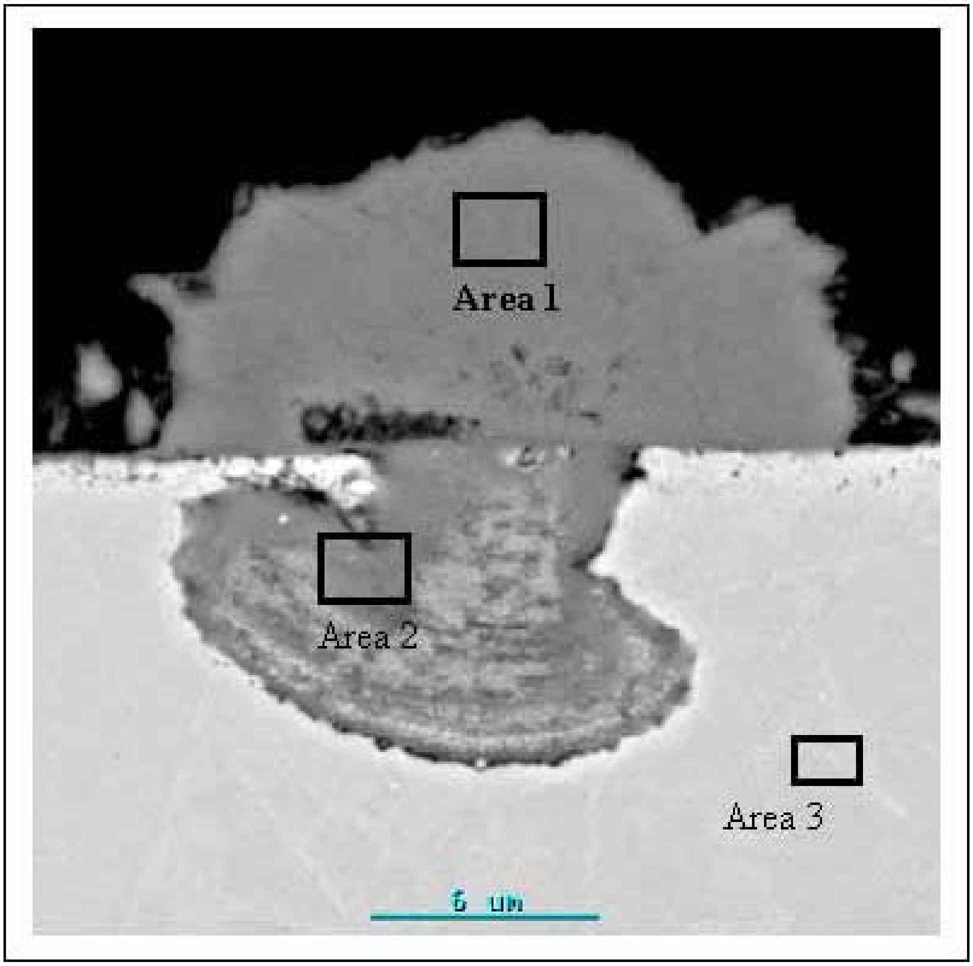


Figure 29: High Magnification SEM Image of a Localized Penetration on the Super 304H Surface (650°C)

Task 8 ASME Code Support Activities (ALSTOM)

The major objectives for Task 8 are:

- Review the methods used by Section I of the ASME Boiler and Pressure Vessel Code to utilize materials properties and behavior models in the design of ultra-supercritical boilers.
- Develop and document methodologies whereby the results of the other tasks within this program may be most effectively applied within the ASME Section I design environment.
- Pursue the incorporation of such methodologies into Section I.

These objectives will be accomplished through execution of seven sub-tasks. Where activity on these sub-tasks occurred during the reporting period, it is described below.

Task 8A: Task Management and Direct ASME Code Activities (ALSTOM)

- Objective: The primary objective of this subtask is the overall management of the task, meetings and any activities required for gaining approval of any necessary ASME Code Cases.

- Progress for the Quarter: A Task 8 meeting was held in Chattanooga on June 13, 2003. The objective of each task was reviewed and refined (see below).

- Concerns: Assignment of responsibility for sub-tasks and distribution of funding.

- Plans for the Next Quarter: Assignment of responsibility of those subtasks not presently assigned and resolution of funding distribution issues.

Task 8B: Material Data Collation and Processing (Unassigned)

- Objectives: The creation of documentation to ensure that quality test data is transferred between tasks and that this data remains traceable. A second objective is the analysis of such data with the objective of improving the statistical correlations.

- Deliverables:
    - Material data transfer sheets.
    - Electronic data repository.
    - Recommendations for statistical analysis of data.
    - Data compendia and fits for each of the key materials (Super304H, IN617CCA, Haynes230, IN740).
    - Code case packages and submissions to code committees.

Progress for the Quarter: Ian Perrin (ALSTOM) has prepared and issued spreadsheets for tensile and creep test data. John Shingledecker of Oak Ridge is looking into the various ways that this data can be made available to all parties.

- Concerns: No lead investigator yet identified.

- Plans for the Next Quarter: The location of the database and access procedures will be specified.

Task 8C: Design Rules (Unassigned)

- Objective: Develop and present to ASME, alternative design rules incorporating the outputs of the other tasks and subtasks.

- Deliverables
  - Overview report drawing together work of the subtasks.
  - Design rules (code case) for unwelded parts.
  - Design rules (code case) for parts with similar metal welds.
  - Design rules (code case) for parts with dissimilar metal welds.

- Progress for the Quarter: There will be no progress on this sub-task until the outputs of other Tasks and subtasks are available.

- Concerns: None

- Plans for the Next Quarter: None

Task 8D: Reference Stress Methods (ALSTOM)

- Objective: Develop and issue a description of reference stress methodology and its application to ASME geometries.

- Deliverables:
  - Topical Report on reference stress methods including compendium of solutions.
  - Example ASME problem showing use of reference stress and comparison with “full” analysis.

- Progress for the Quarter: The draft report has been completed and issued for comments (see “Report Summary” below).

- Concerns: None

- Plans for the Next Quarter: By the end of August this subtask will be complete with the publication of the topical report and the example problem.

Task 8E: Continuum Damage Mechanics (Unassigned)

- Objectives: The objective of this subtask is to analyze uniaxial and multiaxial creep test data from Task 2 for several (three) materials to:

    - establish the continuum damage mechanics (CDM) parameters,
    - evaluate multi-axial strength theories and failure criteria,
    - create deformation and fracture maps.
    - evaluate and compare CDM, reference stress and Omega models of typical ASME geometries.

- Deliverables:
    - Report to summarize data fitting of CDM parameters for physically based and Omega creep models, including multiaxial parameters.
    - Report containing deformation and fracture mechanism maps for each material
    - User material subroutine for use with finite element code (e.g. ABAQUS).
    - Report to summarize the analysis of the validation tests and component simulations, including comparison between approaches (CDM, Omega, reference stress, etc).

- Progress for the Quarter: Definition of objective and deliverables.

- Concerns: No lead investigator yet assigned.

- Plans for the Next Quarter: Selection of lead investigator and initiation.

Task 8F: Weld Analysis and Assessment (ALSTOM)

- Objectives: Create simplified analysis models of welds and heat affected zones utilizing material properties obtained from the open literature and from Task 2.

- Deliverables
    - Topical review of weld analysis and assessment in creep range.
    - Collation of material data for weld metal and heat affected zones.
    - Creep models for weld metal and heat affected zone regions.
    - Report documenting the simulation of welded specimens and common Code geometries.
    - Report documenting the development and use of approximate weld assessment methods.

- Progress for the Quarter: Definition of objective and deliverables.

- Concerns: None

- Plans for the Next Quarter: Initiation by lead investigator.

Task 8G: Basic Design Rules for Unpenetrated Cylinders (ALSTOM)

- Objectives: Review the various equations used by the ASME Code, Section I for Power Boilers to define the minimum thickness of unpenetrated cylinders under internal pressure and develop a single methodology applicable to ultrasupercritical boilers.

- Deliverables
  - Report summarizing existing approaches and comparing and contrasting their predictions.
  - Report recommending a single equation with supporting theoretical data.
  - Code case submission.

- Progress for the Quarter: Definition of objective and deliverables.

- Concerns: Funding distribution.

- Plans for the Next Quarter: Initiation by lead investigator.