Annual Progress Report

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Executive Summary

The overall objective of the project is to contribute to the development of materials technology for use in ultra supercritical (USC) pulverized coal power plants capable of operating with steam up to 760°C (1400°F), 35 MPa (5000 psi). R&D is needed on advanced materials to lead to a full-scale demonstration, and eventual commercialization of USC power plants. The lack of materials with the necessary fabricability, and resistance to creep, oxidation, corrosion and fatigue at the higher steam temperatures and pressures currently limit the adoption of advanced USC steam conditions in pulverized coal-fired plants. A major, five year, national effort sponsored by DOE and OCDO to develop materials for USC boilers has been in progress and is being carried out by a Consortium of EIO, EPRI, Oak Ridge National Laboratory and all the domestic boiler manufacturers. Development of materials technology for USC steam turbines to match the USC boiler conditions is clearly necessary and is a priority in order to support commercialization of USC power plants. With this objective in mind, A 3 year project on USC turbine materials was started by OCDO and DOE with cost sharing and technical participation from, Alstom, EPRI, General Electric and Siemens. Oakridge National Labs was funded by NETL/DOE directly to provide support to the project.

Fig1 shows the basic tasks being addressed and there interrelationship...
Results of Work During Reporting Period

(This is an annual report concerning the progress for the period Oct 2005 to December 2006)

Task 12.1 Coatings for Steam Oxidation and SPE Protection

Sub task A-Alstom Power

Task Objective:
Identify options for coatings that will protect HP turbine components from excessive steam oxidation at inlet temperature up to 1400F (760 C). Additionally, identify coatings to provide control of solid particle erosion of candidate high-temperature alloys equal to or better than existing alloys.

Approach:
The consortium participants will select steam Oxidation Coatings – Candidate alloys. A review of coatings for oxidation prevention, including those used in aerospace applications, suitable for the chosen alloys will be undertaken. A selection of representative coating approaches will be applied to test coupons. The coated test coupons, along with uncoated baseline specimens, will be exposed to atmospheric pressure steam at high temperature for up to 15,000 hours. The coupons will be periodically monitored for weight gain and
occasionally examined metallographically for oxide thickness and penetration depth. Data will be examined to determine oxidation kinetic parameters. Thermal cycling of the coupons will be introduced if deemed necessary for the realistic evaluation of coating performance. Consideration will also be given to the resistance of coatings to Solid Particle Erosion (SPE) but no testing of SPE resistance will be completed; this will be the responsibility of Siemens Westinghouse.

Progress during the Reporting Period:
A test plan was prepared utilizing two steam oxidation test methods to expose specimens to atmospheric pressure steam. Thermogravimetric Analyzer (TGA) testing provides short-term, detailed data on oxidation. Candidate substrate materials have been selected and include: Nimonic 105, Nimonic 263, Haynes 230, Haynes 282, Inconel CCA 617, Inconel 740, and Udimet 720LI. Test substrate materials have been acquired with the exception of Inconel 740 and Udimet 720LI. Coatings have not as yet been selected. Potential coatings candidates include:

- Diffused aluminizing treatments (painted slurry - Sermaloy J or 1515 variants)
- Pack aluminide
- Overlay aluminizing (Sermetal W type) - probably need a seal coat
- HVOF Ni20Cr
- HVOF Cr Carbide Nicer
- HVOF Fe50Cr
- Electroplated Co-Cr Carbide (Tribune 104)
- Chromatin
- Silicon zing
- Plasma nitriding
- PVD CrN + Plasma nitride

Test facilities for each test method were re-comissonned for this program.

The following table provides an overview of the planned exposures for each test method.

<table>
<thead>
<tr>
<th>Temperature - C</th>
<th>Material</th>
<th>Time - h</th>
</tr>
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<tbody>
<tr>
<td>TGA</td>
<td></td>
<td></td>
</tr>
<tr>
<td>700</td>
<td>Nim 105</td>
<td>168</td>
</tr>
<tr>
<td>760</td>
<td>Nim 263</td>
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</tr>
<tr>
<td>800</td>
<td>Haynes 282</td>
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<td></td>
<td>Udimet 720 LI</td>
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<td></td>
<td>In 740</td>
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<td></td>
<td>Haynes 230</td>
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<td></td>
<td>CCA 617</td>
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<td>Long Term Exposures</td>
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<tr>
<td>700</td>
<td>Nim 105</td>
<td>500</td>
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<tr>
<td>760</td>
<td>Nim 263</td>
<td>1000</td>
</tr>
</tbody>
</table>
Table 1: Outline of steam oxidation test parameters.

TGA exposure testing has commenced. The 800 C and the majority of the 760 C tests have been completed for the available test materials.

Sub Task B-Siemens
Objective:
The objective of this sub task is to identify coatings resistant to solid particle erosion under conditions simulating those in a USC turbine

Approach:
Substrates and coatings will be selected in consultation with the consortium partners and will be tested in a furnace under erosive conditions

Progress to date
This section of the program involves the selection of erosion coatings and their testing in an environment as close as possible to that predicted to be found in a USC turbine. Initial screening coatings have been selected, and Siemens is currently in negotiation with NETL Albany as to the exact test to be carried out. The base substrate to be used has been narrowed down to two alloys, Udimet 720LI and Inconel 740. Limited substrate testing of Udimet 720 is planned to be carried out within the Welding section of this program, and the results will be compared with those of the newer IN 740 material, which is being tested here. A limited amount of material testing is being carried out within this section of the program on the newest variant of IN 740. The primary reason for this is to identify the optimal heat treatment and to examine the long term stability of the alloy at the temperature of use. Material is being tested using the baseline 1120°C-1hr + 800°C 4hr heat treatment developed for the material as well as a limited number of tests on unheat-treated material as well as material treated at a higher solution temperature. To date creep rupture data is available. Whilst most samples are still under test, preliminary conclusions can be drawn. From a creep perspective, the standard aging treatment does not appear to significantly improve the properties as opposed to solution without age. Raising the solution temperature appears to increase the creep strength, though more data will be required to confirm this. The standard heat treated data appears to lie within the bound of the data as estimated by ORNL, but on the lower side of the mean.
Task 12.2 Welded Configuration for HP and IP Rotors

Sub Task-A: Alstom
Objective:
To establish component requirements and candidate materials for welded rotors;
To perform basic welding research to identify issues and best approaches for welding precipitation strengthened nickel alloys;
Create test welds and perform metallurgical and mechanical testing of welds.

Approach
A preliminary conceptual design study will establish the typical component dimensions and service conditions. Candidate materials will be reviewed and information on their weldability collated, drawing on experience welding similar materials in Gas Turbine applications. Having identified candidate alloys and joint types, basic welding process development will begin for a precipitation strengthened nickel alloy. Such development would likely include techniques such as varistrain testing to assess susceptibility for cracking. Based on the knowledge gained and existing experience with solution strengthened nickel based alloys, trial welds will then be created and inspected nondestructively and destructively to assess their metallurgical characteristics. Mechanical testing will be used to evaluate the tensile and creep strength of sample welds.

Progress during the Reporting Period:
The philosophy for achieving a welded rotor construction was formulated
The development of a welded rotor for the proposed USC cycle will require the joining of a precipitation hardened nickel alloy with a solution hardened alloy. Consideration of the available information on candidate welded rotor materials lead to the decision to base the initial study of welding fundamentals on the requirements for joining Haynes 263 with Inconel CCA 617. A study scope was prepared in concert with the Edison Welding Institute and a proposal was received from them to execute this 8-month study. A final deliverable of the program will be a welded joint between 2-inch thick sections of the selected materials. A purchase order has been placed with EWI. An order was also placed for the required Haynes 263 alloy. Suitable Inconel CCA 617 was available from studies in the DOE Boiler Materials program.

Sub Task B: Siemens
Objective:
To increase the operating temperature of the rotors, Siemens intention is to form a fabricated rotor consisting of conventional steel at the cooler sections (below 600°C) and Nickel-based superalloys at the higher temperatures. The EIO program objective is to build upon this to add higher temperature capable stages on the front of the 700°C construction.

Approach
Candidate alloys will be selected, test welds will be made and subjected to mechanical tests. Siemens will use its gas turbine rotor welding experience to weld higher strength materials to form a composite alloy rotor. Joints are to be developed to allow the joining of two further high temperature alloys allowing temperatures of approximately 730 and 760°C. Design analysis has shown that the use of a ‘diagonal vane stage’ which accelerates and cools the steam can reduce the RIT (rotor inlet temperature) to approximately 730°C with relatively low efficiency losses.

Two weld geometries will be considered for each joint using filler-less electron beam welding, and the decision as to which will be selected will be determined by the mechanical behavior of the weldments. For design A, we will carry out 15-20 welds of the Haynes 282 to U720LI joint and 8-10 welds for the Haynes 282 to IN617 joint.

The process will aim for a 21 mm penetration on 25 mm thick plates. After metallography of the test pieces and simple mechanical tests, the optimal process will be selected and further samples manufactured for mechanical tests. Once this process has been optimized, the second geometry will be welded.

![Figure 1. Schematic of EB weld trial geometry, Design A](image-url)
Figure 2. Schematic of EB weld trial geometry, Design B

Should funding permit it, ring demonstration parts will be produced with the selected geometry and weld parameters.
Concurrently, baseline material testing of the three alloys will also be carried out to allow a direct determination of the weld strength deficit. Tests will consist of creep, fatigue and tensile tests at 700, 750 and 800°C.

Progress To Date
Candidate materials have been identified and a test program has been formulated. Materials have been ordered. A detailed selection process was carried out to select materials capable of being operated at either 730 or 760°C for 100,000 hrs. From this process, two alloys were selected, namely Haynes 282 a new hybrid solution-precipitation strengthened alloy from Haynes Intl and a high strength gas turbine rotor alloy Udimet 720LI. Detailed discussions were held with the forging houses as to their capabilities of forging Udimet 720LI into sufficiently large disks. It was concluded that conventional forging of the required geometry was realistic, but if necessary isothermal forging or powder-formed ingots could be considered. This development is outside of the scope of the EIO program. The risks incurred in forging Haynes 282 were considered to be low due to the slow precipitation kinetic characteristics of the alloy, which allow a large degree of deformation to occur without severe hardening.

The joint configurations under consideration are IN 617 to Haynes 282 and Haynes 282 to Udimet 720LI. The first of these is not considered to be a high
risk, but Udimet 720LI is a material that is known to be a challenge from a welding viewpoint. The fallback position should this be unsuccessful is to use the diagonal stage construction to avoid the need to use this material.

Haynes 282 will be used in the conventionally forged and heat treated condition, but to reach 760°C, Udimet 720LI will have to undergo a complex heat treatment to provide sufficient mechanical properties to meet the Siemens requirements. As the alloy was initially developed as a blading material, the high creep strength super-solvus heat treatment will be used for the disk rim and the more recently developed sub-solvus disk treatment will be needed for the rest of the rotor. Optimizing the forging and heat treatment parameters is outside the scope of this program.

Due to procurement problems, weld development has not started within the program. Of the three alloys required, 2 are scheduled for delivery in December 2006 and the third in January 2007. Weld trials are scheduled to start in February 2007.

Task 12.3 Non-Welded Integral Rotor Development
General Electric

Objective:
Identify HP rotor alloys for 760C/35 MPa steam conditions

Approach:
Based on literature survey, Candidate alloys that meet certain criteria will be selected and evaluated using a variety of mechanical tests. These criteria include as a minimum, 100000hr rupture life at 1400F(760C) and 100MPa(15Ksi), 50ksi nradial yield strength at 1400F and a room temperature fracture toughness of 50 KSI Square root of inch.

Progress to date
Based on an extensive survey of literature, a spreadsheet of mechanical, physical and thermo physical properties has been created for 19 candidate rotor alloys. ThermoCalc simulations were performed on all candidate alloys, and microstructural stability was assessed based on precipitate, carbide and TCP phase formation, solvus temperatures, processing windows, etc. Heat transfer simulation, of a large IP rotor ingot under air cooling, and fan cooling was evaluated; The core cooling rate was found to be half that of the surface, and therefore the likelihood of cooling in air, without cracking was ascertained. Based on available mechanical property and microstructural stability information, the following alloys were selected for further study: Nimonic 105, Udimet 720LI, IN740 and Haynes 282. The various phases of alloy selection, heat treatment and mechanical testing are described below.
Material Procurement and Heat Treatment

Haynes 282

The Haynes 282 was received in the solution-annealed condition. Tensile, LCF and creep tests were performed in this condition to establish base-line mechanical behavior against which the effects of age-hardened and overaged microstructures could be compared.

Age-Hardened Condition

1. Heat to 1850F and hold at this temperature for 2 hours.
2. Air cool to between 1400 and 1450F, or any convenient temperature below 1450F.
3. Reheat/heat to 1450F and hold at this temperature for 8 hours.
4. Air-cool to room temperature.

Overaged Condition

Heat-treat the age-hardened Haynes 282 at 1425F for 250 hours: furnace cool when done.

Nimonic 105

The Nimonic 105 was received as cold drawn bar. A section of the bar was used to determine base-line tensile, LCF and creep properties. Subsequently, the bar was heat treated to peak strength and then overaged according to the following schedules.

Age-Hardened Condition

Two-Step Super-Sub Solvus Solution Heat Treatment

a) Preheat to 1925F in vacuum and hold for ½ hour.
b) Heat to 2125F in vacuum and hold for 4 hours.
c) Furnace cool to 2030F in vacuum and hold at temperature for 4 hours followed by fan cooling to RT.
d) Heat to 1475F and hold for 24 hours followed by air-cooling.

HT-Overage Schedule

Heat-treat the two-step super solvus solution heat-treated material at 1450F for 250 hours, furnace cool when done.
Udimet 720Li

The Udimet 720Li was received as two forged disks, each approximately one inch thick. The disks were heat-treated according to the following schedule:

a) Preheat to 1925°F in vacuum and hold for ½ hour.
b) Heat to 2025°F in vacuum and hold for 4 hours.
c) Nitrogen quench in vacuum, then reheat to 1200°F in air. Hold for 24 hours then quench in oil.
d) Reheat to 1425°F in air and hold for 16 hours. Air cool to room temperature.

Tensile, LCF and creep tests were performed in this condition to establish baseline mechanical behavior. No additional heat treatments were done to this alloy due to insufficient material.

Mechanical Properties

Tensile Behavior. The UTS values are summarized in Figures 1 for Haynes 282 SA, Nimonic 105 AP (as processed) & HT (age hardened for strength), and Udimet 720Li HT. (Preliminary testing is still in progress for the overaged Nimonic 105 and Haynes 282, and age hardened Haynes 282.)

![Figure 1. UTS as a function of temperature for Haynes 282 SA, Udimet 720Li HT, Nimonic 105 AP and Nimonic 105 HT.](image-url)
From Figure 1, it is clear that as the test temperature increases to 1500F, the UTS of these alloys converge. However, at 1400F, Udimet 720Li HT possesses the highest UTS with the Nimonic 105 alloys about midway between Udimet 720Li and Haynes 282. It is interesting that Haynes 282 shows a little “bump” in strength in the range of 1200-1400F.

Figure 2 shows the results for 0.2% YS. Once again, the values for 0.2% YS converge for these three alloys at 1500F, and again at 1400F, the alloys rank in the same way as UTS. Again, Haynes 282 SA shows a bump in strength between 1200-1400F.

At this point, the only tests outstanding are those for Haynes 282 age hardened for strength and the Haynes 282 and Nimonic 105 (age hardened condition) heat-treated to produce an overaged precipitate structure.

The results of this study will provide a good indication of the relative tensile behavior of these alloys for different processing conditions, allowing a rational choice of processing options for large-scale ingots subject to slow cooling rates. Of special note will be the results of those alloys with an overaged precipitate structure.
Creep Behavior. Creep tests are underway. Based on 0.2%YS & UTS, strength levels thought appropriate for the alloy were selected to give reasonable creep rupture times (up to about 7,000 hours). Figures 3 and 4 show creep rupture data for Nimonic 105 AP and Udimet 720Li. So far, four creep specimens have failed for the Nimonic 105 AP and an approximate parameterized curve has been included in the figure. (Selected Nimonic 105 HT specimens have been added to the test program and those tests are underway.)

![Graph showing creep rupture data for Nimonic 105 and Udimet 720Li.](image)

**Figure 3. Parameterized rupture for Nimonic 105: failed and in progress.**

The creep testing program for Udimet 720Li is complete. The starting stresses were high enough to cause failure in all bars in well under 1,000 hours, surprising given the 0.2% YS of these alloys. This material will have to be examined in more detail at lower stresses to see if creep rupture behavior is commensurate with that of the Nimonic 105 and Haynes 282 alloys.

The Haynes 282 alloy, both SA and HT, are in test. Creep times are approaching 3,000 hours and results are exceeding expectations, given the much lower 0.2% YS of the alloy. One point of note: The starting stresses for the Haynes 282 are much lower than for the Nimonic 105 and Udimet 720Li.

**LCF Behavior.** Due to increased backlog at the vendor only one set of 20 cpm LCF tests have been completed to date for the Nimonic 105 AP. Run out is set at 100,000 cycles. The results of these tests are shown in Figure 5. Two tests were
run at each strain level. A curve of “best fit” was determined and overlaid on the data points.

\[ LMP = [T + 460][20 + \log t] \]

\[ \text{Stress (y)} = 1853 - 72.8 \text{LMP} + 0.7 \text{LMP}^2 \]

R\text{2} = 0.98

Figure 4. Parameterized rupture for Udimet 720Li: all testing completed.

Figure 5. 20 cpm LCF at 1400F for Nimonic 105 AP.
Summary

The preliminary screening of 1400F alloys is nearing completion for tensile behavior. Test programs for 20 cpm LCF have been finalized with specimens prepared. Samples are at the vendor for testing. Creep testing is underway for all alloys and conditions.

Although not discussed in this report, microstructural examination of the alloys in the various conditions is also underway. Results will be forthcoming.

Results for 0.2% YS look promising with values above 60 ksi at 1400F. The 20 cpm LCF tests on Nimonic 105 AP at 1400F are encouraging as well, with run out occurring at a strain level of 0.50%. Creep testing is still underway but preliminary results for Haynes 282 (low stress levels) and Nimonic 105 look promising.

Task 12.4 Castings :
Siemens

Objective:
The objective of this task to identify casting materials capable of operating at 1400F.

Approach:
Extensive survey of literature was carried out

Progress to date
This section of the program is to select and determine appropriate materials for casings to operate at up to 760°C. A selection procedure with a wide range of CTQs was carried out during the year, but ran into problems. Two alloys were initially selected based on the potential that their mechanical properties in an air-cast form would reach those needed. The two alloys selected, Haynes 282 and IN 740 have mechanical properties in the wrought form that easily reached the targets. Once the manufacturers of the two materials were contacted, it became apparent that we would be unable to work with them. Haynes international were willing to provide material, but only in 20,000 lb heats. Special Metals were unwilling to provide IN740 for casting. To be castable, the silicon content of the alloy would need to be reduced, however this would have meant that the composition was outside of their patent. As a consequence, the analysis of the two materials was abandoned. The selection process is currently being re-evaluated to identify two further alloys capable of meeting the requirements.

TASK: 12.5 Design and Economic Studies
Objective:
To assess the cost and performance tradeoffs associated with the use of expensive nickel-based alloys and integrate understanding developed during the project to ensure a coherent vision is developed for USC steam turbines

Approach.
Based on design studies performed within this task and other task, some of the cost/performance tradeoffs (e.g. relax pressure to reach higher temperatures, cooling, etc.) will be examined and USC plant models will be used to establish costs. [For example, in the USC boiler project, a task ascertained that based on the increased cycle efficiency the capital cost of the boiler could increase by a certain percentage and still provide an economic plant.] The impact of a USC steam turbine on such plants needs to be quantified. This will require some up-front work and additional work toward the end of the project when the initial studies can be revisited based on what has been learnt. This will ultimately lead to an assessment of the benefit provided by USC steam turbines and a vision/roadmap for their future development and deployment. It is envisaged that some associated studies would also be performed to establish the cycling capability of USC turbines since this may affect their market acceptance and economic evaluation.

Progress during the Reporting Period:
Initial review of the results to date of the DOE USC Boiler Materials program and other information relative to high temperature materials led to a reevaluation of cycle pressure and temperature parameters. Consideration of acceptable boiler conditions leads to the following tentative cycle steam conditions:

- Main steam 348 - 354 bar, 732 C
- Reheat pressure 70- 60 bar, 760 C
- Boiler pressure drop 41.5 - 48.3 bar (excludes pressure drop in piping and fittings to and from the boiler)
- Desired economizer inlet temperature 304 - 315.6 C. Higher feedwater temperature is feasible and should be investigated, but its impact on boiler efficiency, surfacing, and waterwall materials might be a problem for some coals
- Condenser pressure of .085 bar, consistent with previous steam turbine heat balances.
- Steam turbine heat input at two full load (MCR) conditions: 5,503.4 x 10^6 kJ/hr and 6,446.3 x 10^6 kJ/hr.

The above values represent the upper range of the acceptable combination of steam pressure and temperature. The next step will be preparation of steam turbine heat balances for MCR, 70%, 50% and 30% loads for the lower heat input case and at MCR only for the higher heat input case.
A 760 C / 287 bar cycle has been promoted in the U.S. A comparison of the thermal efficiency of this cycle will be prepared to determine to examine the relative merits of lower pressure.

TaskX-Contributions by ORNL

Following the Kick-Off meeting for the DOE/OCDO USC Materials Consortium - Steam Turbine Group, held Nov. 21, 2005 in Washington, DC, ORNL attended a joint USC Boiler and Turbine Group Steering Committee Meeting at EPRI in Charlotte, NC, on January 18-19, 2006. This meeting coincided with a parallel meeting of the EPRI Coal Fleet. ORNL provided input on alloy selection for USC steam turbine applications, based on current steam oxidation behavior and mechanical properties testing (mainly data of Shingledecker, ORNL, USC Boiler Consortium), including existing commercial Ni-based superalloys, and new alloys being introduced or developed by industry. ORNL summarized its latest data on cast CF8C-Plus austenitic stainless steel as a potential turbine casing material, its summary of U.S. utility interest in the DOE/FE USC Materials program, and a perspective from European utilities. Discussion focused the turbine consortium interest on cast Ni-based superalloys for casing applications, and interest on the latest data on the newest alloys.

In March, ORNL reviewed the materials selection datasheet distributed by General Electric (GE) (J. Hawk), as follow up to action items from the USC Steam Turbine Group Steering Committee meeting on January 18, 2006 at EPRI in Charlotte, NC. ORNL (J. Shingledecker) added mechanical properties and microstructural information to that spreadsheet for alloys CCA617, 617, Haynes 230, Haynes 25 and Inconel 740, and sent it back to GE for incorporation into the master datasheet. GE also contacted ORNL with a request for potential materials for screening tests. Due to the collaboration between USC Boiler and Turbine groups at the Charlotte meeting, ORNL provided the relevant contact information for other members of USC Boiler group (who have agreed to provide extra materials, where available), because ORNL does not have extra material.

Earlier in May, John Shingledecker (ORNL) interacted with Doug Arrell (Siemens) on details of heat compositions, heat-treatments and ORNL creep data on alloy 740, based mainly on his work for the USC Boiler Consortium. Phil Maziasz (ORNL) contacted and discussed alloy selection and data needs with each of the USC Steam Turbines Consortium principles. As noted in the previous OCDO Project Status Report, there is clearly a consensus focus on the Nimonic 105, Haynes 282, and Udimet 720Li alloys, as well as a castable version of Special Metals 740, or possibly an alloy with composition between alloys 740 and 263.

ORNL (Phil Maziasz) presented a talk entitled “Advanced Materials for USC Steam Turbines” (co-authors: Ian Wright and Vis Viswananthan (EPRI)) at the Session on Advanced Materials, chaired by Fred Glaser (DOE/HQ), at The 2006 Clearwater Coal Conference, held 21-26 May, 2006 in Clearwater, FL.
ORNL attended the Steering Committee Meeting held for the DOE USC Steam Turbine and Boiler Consortiums in Cleveland, OH on August 30, 2006. ORNL presented its new plans for FY2007, including 3 different tasks that best utilize ORNL’s experimental capabilities and expertise. Task 1 included long-term materials performance and degradation testing of wrought Ni-based superalloys at the USC conditions. Task 2 included evaluation and development of Ni-based superalloy castings capable of use for the turbine casing. Task 3 involved evaluating and testing coatings in the ORNL steam rig relevant to Tasks 1 and 2. After the meeting, follow-up discussions of mutual interest were held with PI’s at Alstom and at General Electric.

Concerns
ORNL efforts throughout 2006 were marginalized due to lack of DOE/FE funding. ORNL received no new FY2006 money, and the carry-over funding from last year was exhausted after the joint USC Boiler and Turbine group's meeting in January, 2006. ORNL therefore currently remains unable to perform any significant work.