OXIDE DISPERSION-STRENGTHENED HEAT EXCHANGER TUBING

Vision 21 Program Review Meeting
November 6-7, 2001
Morgantown, WV

Mark A. Harper, Ph.D.
Special Metals Corporation
Huntington, WV

ABSTRACT

Oxide dispersion strengthened (ODS) alloys (e.g. the INCOLOY® MA956 alloy) are known for their excellent high temperature properties and are prime candidate materials for the construction of very high temperature heat exchangers that will be used in Vision 21 power plants. The main limitation of these materials is their poor weldability. Commercially available ODS tubing also tends to exhibit relatively poor circumferential creep strength due to current processing practices resulting in a fine grain size in the transverse direction. Thus far, these two characteristics of the ODS tubing have restricted its use to mostly non-pressure containing applications.

The objectives of this program are to develop:

(a) an MA956 tube with sufficient circumferential creep strength for long term use as heat exchanger tubing for very high temperatures;
(b) a welding technique(s) for producing adequate joints between an MA956 tube and an MA956 tube, and an MA956 tube and an INCONEL 601 tube;
(c) the bending strain limits, below which recrystallization will not occur in a MA956 tube during normal operation; and
(d) the high temperature corrosion limits for the MA956 alloy with respect to working-fluid side and fireside environments.

Also, this program seeks to generate data for use by heat exchanger designers and the ASME Boiler and Pressure Vessel Code, and perform an analysis of the mechanical property, tube bending, and corrosion data in order to determine the implications on the design of a very high temperature heat exchanger (T>1093°C/2000°F).

After one year, work is currently being conducted on increasing the circumferential strength of a MA956 tube, developing joining techniques for this material, determining the tube bending strain limits, and establishing the high temperature corrosion parameters for the MA956 alloy in environments expected to be present in Vision 21 power plants. Work in these areas will is continuing into the next fiscal year, with success anticipated to produce innovative developments that will allow the reliable use of ODS alloys for heat exchanger tubing, as well as a variety of applications previously not possible with metallic materials.

® INCOLOY and INCONEL are registered trademarks of Special metals Corporation

INTRODUCTION

The Department of Energy (DOE), National Energy Technology Center (NETL), has initiated a strategic plan for the development of advanced technologies needed to design and build fossil fuel plants with very high efficiency and environmental performance. These plants, referred to as "Vision 21" by DOE, will produce electricity, chemicals, fuels, or a combination of these products, and possibly secondary products such as steam/heat for industrial use. In addition, this program will achieve radical improvements in the performance of existing power technologies and seek to virtually eliminate the environmental concerns associated with the use of fossil fuels.

Interest in increasing the efficiency of coal-fired power plants has led to the examination of alternatives to the steam boiler-Rankine cycle systems, for which increases in efficiency have been limited by both the slow progress to be able to handle steam at temperatures above 565°C (1050°F) and the unavailability of easily-accessible sources of naturally-occurring low-temperature cooling water. Indirect-firing of gas turbines in open or closed cycles is one approach to linking the higher efficiencies possible via the Brayton cycle while still using coal as the fuel. An experimental program in the 1980's[1] demonstrated a coal-fired, low-emissions heat exchanger (fluidized-bed combustor) capable of heating air to 843°C (1550°F) in a metallic heat exchanger, and to 954°C (1750°F) or 1232°C (2250°F) with an additional ceramic heat exchanger. Current programs involving indirectly-fired gas turbine cycles are aimed at high cycle efficiencies, of the order of 47 percent based on the higher heating value (HHV) of the fuel, and involve open cycle systems in which air is heated to 760°C (1400°F) in a metallic heat exchanger, followed by further heating to 982°C (1800°F) in a natural gas-fired ceramic heat exchanger[2-4]. A variant of this approach is where part of the coal is pyrolyzed to produce the fuel gas used to fire the ceramic heat exchanger or the turbine with air entering the turbine heated to 1288°C (2350°F). A further program envisions using a coal-fired ceramic heat exchanger for the whole duty of heating air to 1200°C (2192°F)[5].

Successful implementation of indirectly-fired cycle technologies will require the development of a durable coal-fired heat exchanger capable of heating the working fluid to very high temperatures, in addition to adapting a gas turbine for this particular duty. The primary functions of the heat exchanger material will be to contain the working fluid at the maximum temperature and pressure consistent with the turbine design and provide sufficient resistance to the working fluid and fireside environments in order to give an acceptable component lifetime. The heat exchanger material must also possess adequate fabrication properties to allow for traditional methods of heat exchanger production or the technology must be developed to allow for fabrication using non-traditional methods.

This project is seeking to develop a MA956 heat exchanger tube which will lead to the design and fabrication of a MA956 full-scale tube heat exchanger composed of the referenced alloy. The alloy MA956 is an oxide dispersion strengthened (ODS) material that possesses superior creep strength and corrosion resistance at very high temperatures (e.g. T > 2000°F) compared to traditional wrought or cast alloys. However, the creep properties are unidirectional (typically stronger in the longitudinal direction compared to the transverse direction), fabrication of components made from this alloy is relatively difficult, and the corrosion limits of the alloy MA956 in coal-fired environments are not known. Thus, the technical tasks being executed in this Vision 21 project are:

Task 1: Project Management
Task 2: Improvement of Circumferential Creep Strength of MA956 Tubes
Task 3: Joining
Task 4: Bending of MA956 Tubes
Task 5: High Temperature Corrosion Limits of MA956
Task 6: Generation of Data for Designers
Task 7: Implication of ODS Properties on Heat Exchanger Design
Task 8: Reporting

The members of the team conducting this research are: Huntington Alloys (HA), Jeffrey Blough at Foster Wheeler Development Corporation (FWDC), Ian Wright at Oak Ridge National Laboratory (ORNL), Bimal Kad at University of California, San Diego (UCSD), Marvin McKimpson at Michigan Technological University (MTU), and Larry Brown at the Edison Welding Institute (EWI).

EXPERIMENTAL PROCEDURE

Experimental work associated with the tasks identified in the previous section are discussed below.

<u>Task 2: Improvement of Circumferential Creep Strength of MA956 Tubes</u>

In order to understand the effect of extrusion + thermomechanical processing parameters on the microstructure of the MA956 tube, the first phase of this project will result in the matrix of tests shown in Table 1 below to be performed at HA, with analysis of the resulting microstructures conducted at UCSD.

Table 1
Matrix of Extrusion + Cold Work + Recrystallization Parameters

Extrusion Temp (°C)	Extrusion Ratio	Amount of Cold Work (%)	Recrystallization Temp (°C)	Recrystallization Time (h)
1000	10:1	0	1000	0.5
1075	16:1	10	1150	1
1150	20:1	20	1300	6
1200		30		
		40		

Also, creep testing is underway at ORNL for the purpose of determining the “stress threshold” curves of the MA956 alloy.

Task 3: Joining

Studies to join MA956 tubing to itself and to a wrought heat-resistant alloy (INCONEL alloy 601) using friction welding, explosive welding, and magnetic impulse welding are being performed by EWI. Also, joining of this material using transient liquid phase bonding is being studied at MTU.

Task 4: Bending of MA956 Tubes

In an effort to determine the bending limits of MA956 tubing such that recrystallization of the alloy does not occur during normal operation, FWDC will be subjecting tubes of this material to 5, 10, 15, 20, and 25% strain, followed by exposure at 2200°F for 100 hours. Analysis of the tube microstructure before and after the exposure will be conducted in order to determine the maximum amount of strain that can be tolerated by a MA956 tube without recrystallization occurring during operation.

Task 5: High Temperature Corrosion Limits of MA956

Complementary laboratory and field exposures are being performed in environments expected to be encountered by the external and internal surfaces of MA956 tubes in service. With respect to the working-fluid side of the tube wall (i.e. the I.D.), laboratory exposures in air are being be conducted at ORNL in order to measure kinetic data and determine the criteria (such as aluminum consumption, change in oxidation rate, and initiation of scale spallation) that would signify the end of service life as governed by oxidation resistance. Regarding fireside corrosion of the MA956 alloy, both laboratory and actual field testing will be conducted by FWDC in order to study the effects of various synthetic flue gases and deposits that are expected to be encountered in Vision 21 plants. In laboratory exposures, data will be obtained and analyzed in order to determine the kinetics, failure criteria for the MA956 alloy, and to develop a wastage prediction model for the material in each gas and deposit environment. The field exposures will use air-cooled exposure probes in an actual-coal fired combustion system to generate 10,000-hour data on the corrosion resistance to the fireside environment. Also, the air-cooled probes will expose the MA956 tube samples to prototypical boiler conditions with atmospheric air on the working fluid side.

Task 6: Generation of Data for Designers

Property data that is being generated in Tasks 2, 3, 4, and 5 will be assembled by ORNL and supplied to designers.

Task 7: Implication of ODS Properties on Heat Exchanger Design

Thermal and structural modeling of MA956 heat exchanger tubes will be performed at FWDC to determine the effects of non-uniform mechanical properties. General-purpose finite element analysis codes, including Abaqus and Algor, will be used for the analyses. Tubes will be evaluated in typical heat exchanger arrangements, subjected to thermal and mechanical loads. It may also be necessary to review vibration characteristics of the heat exchanger tubes. A fluid flow analysis will help define the extent of fluid-elastic vibration in typically supported tube heat exchanger arrangements. The tube response will be described in mode shape plots for natural frequencies. This will help to define additional considerations in the stress analyses. This work will reveal design considerations, accounting for the axial and hoop stresses of MA956 heat exchanger tubes subjected to multi-axial loading. The result will be a set of design guidelines to be followed by the heat exchanger designer. These guidelines will allow designers to more readily incorporate the MA956 tubes in various designs, and accelerate commercialization of this technology. Depending upon the outcome of this task, additional future work (i.e. fatigue and creep testing) may be recommended.

RESULTS AND DISCUSSION

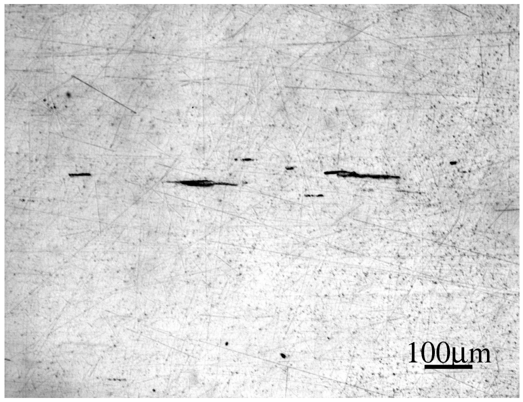
Task 2: Improvement of Circumferential Creep Strength of MA956 Tubes

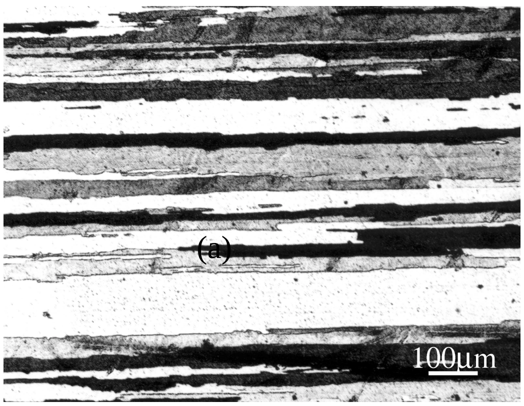
Referring to Table 1 in the previous section and the matrix of tests to be performed at HA, Table 2 shows the work completed to date. At shown in this table, relatively few of the final microstructures have been analyzed. However, analysis thus far does show trends between processing parameters and the final microstructure do exist. For example, regarding the effect of recrystallization temperature, samples annealed at 1000°C did not exhibit any recrystallization, independent of the amount of cold work imposed on the sample. However, samples annealed at 1150°C did show the onset of primary recrystallization with the amount being dependent on the amount cold work. When annealed at 1300°C, all the samples exhibited primary and secondary recrystallization, with the final recrystallized grain morphology being a function of the cold work for a given sample. Figure 1 shows the variation in microstructure morphology observed as a function of cold work for samples annealed 1300°C for six (6) hours. Although sufficient data is not yet available to allow for an understanding and prediction of the effect of the various production variables on the final component microstructure, this knowledge will be critical in producing a tube with the desired microstructure and thus desired mechanical properties.

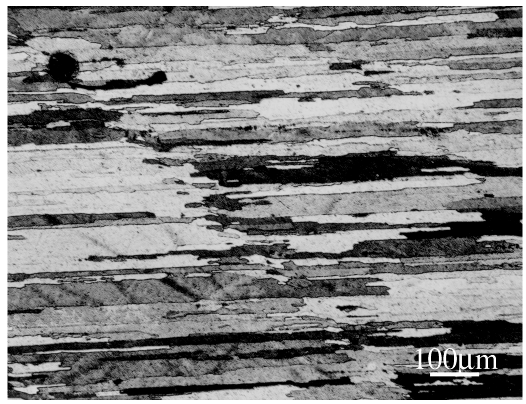
Table 2
Work Completed in Task 2

Operation	Number Required	Number Complete	% Complete
Extrusion	180	180	100
Decanning	540	354	66
Cold Work	540	223	41
Annealing	540	175	32
Microstructure Analysis	540	54	10

(a)







(b) (c)

Figure 1. Variation in microstructure for MA956 rods extruded at 1000°C using a 20:1 extrusion ratio followed by (a) 0% CW, (b) 10% CW, and (c) 30% CW, and then annealed at 1300°C for 6 hours.

Results of the creep testing of specimens cut in the axial direction from the walls of a nominal 1 inch diameter MA956 tube (Heat # WBD0643) are shown in Figure 2. As shown on this Larsen-Miller plot, the data being generated in this program is in agreement with data in the literature. The next step in this task will be to generate data from the transverse direction of the tube.

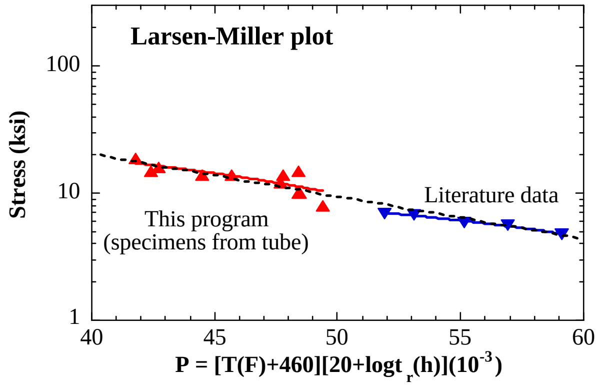


Figure 2. Larsen-Miller plot showing the good agreement between the stress rupture data obtained from this program and stress rupture data obtained from the literature.

Task 3: Joining

Friction welding: Friction welds have been made on the MA956 to MA956 tubing with some of the joints showing tensile properties approaching those of the base material. However, the results to date are not consistent and more work is needed. Also friction welds between MA956 tubing and 601 tubing have been performed with the mechanical properties of such a weld shown in Table 3 below. As shown in this table, the joint strength approaches that of the 601 alloy and thus friction welding shows promise as a suitable technique for joining MA956 to a traditional wrought heat resistant alloy. However, the very low ductility of these joints also shows more work is needed in this area also.

Table 3
Mechanical Property Data for the MA956
Tubing Joined to 601 Tubing Using Friction Welding

Sample Identification	Ultimate Tensile Strength (ksi)	Yield Strength (ksi)	Elongation (%)
MA956/601-1	39.6	37.6	< 1
MA956/601-2	26.4	19.8	< 1
MA956/601-3	43.2	42.4	< 1
MA956/601-4	40.5	40.3	< 1
601*	107.5	42.1	47

* Typical properties of 601 hot finished bar solution annealed at 2000°F.

Transient Liquid Phase Bonding: Boriding work has resulted in the successful production of samples with uniform borided layers 3 to 5 microns thick, and three sets of samples were prepared for joining using hot isostatic press (HIP). These included recrystallized/recrystallized, non-recrystallized/recrystallized, and non-recrystallized/non-recrystallized samples of the MA956 alloy, where both pieces in the bonding couple had been borided, as well as 3 bonding couples with only one borided piece. The joining of the non-recrystallized to recrystallized material was performed in order to cause grain growth across the interface during the joining process. Initial joining runs have resulted in joints that appear void-free with a few residual particles and limited grain growth across the interface (see Figure 3 below). Work continues on refining the boriding technique and additional joining trials using a vacuum hot press are planned.

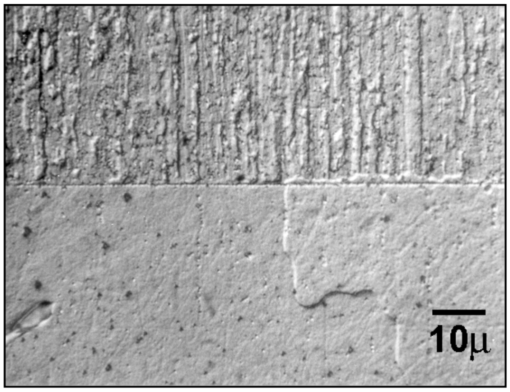


Figure 3. Joint produced using transient liquid phase bonding.

Explosive Welding: Two samples from MA956 plate explosion welded to MA956 plate, and two samples from MA956 plate joined to 601 plate were extracted for metallographic analysis and shear testing. Figure 4 shows the interface of the MA956/MA956 and MA956/601 welds after a post explosion weld heat treatment at 1000°C for 1 hour. As shown in these micrographs, the wave-shaped interface characteristic of a successful explosion weld is present. Table 4 shows the results of the shear testing performed on the as-welded samples and the post weld heat-treated samples. The heat-treated MA956/MA956 sample showed a slight decrease in bond strength as a result of the heat treatment whereas the MA956/601 sample showed an increase in shear bond strength after the heat treatment. However, the measured shear strength for the heat treated MA956/601 sample may be artificially high due to the relatively high ductility of the annealed 601 material. The next step will consist of weld trials on the tube materials of the MA956 and 601 alloys using nearly identical explosion welding parameters as for the plate samples.

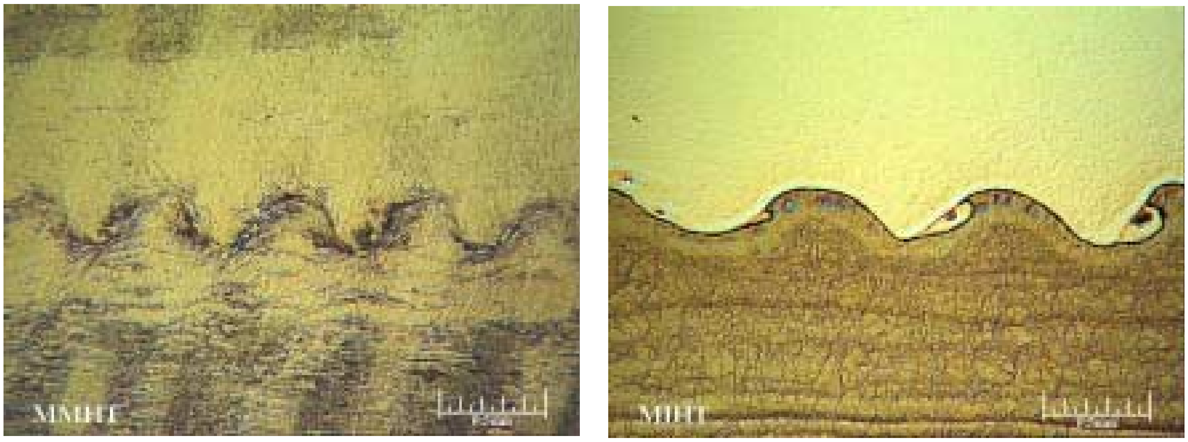


Figure 4. Optical micrographs of the interface of an explosion weld between (a) MA956 and MA956 plate and (b) MA956 and 601 plate.

Table 4
Shear Test Results of Explosion Welds Made on
MA956 plate to MA956 plate and MA956 Plate to 601 Plate

Sample	Shear Strength (ksi)
MA956/MA956	82.0
MA956/MA956 + Heat Treatment*	77.2
MA956/601	79.5
MA956/601 + Heat Treatment*	88.7**

* Heat treatment performed at 1000°C for 1 hour
** Value may be artificially high due to ductility of the 601 after the 1000°C/1 hour heat treatment

Magnetic Impulse Welding: Successful welds using this technique have not been accomplished as of this date. A new machine with four times (4X) the energy of the present machine, which will allow for greater energy input into the joining process, will be installed and operational in November.

Task 4: Bending of MA956 Tubes
In an effort to determine preliminary tube bending limits for the MA956 alloy, tube-flattening tests were performed on recrystallized and non-recrystallized material. The results of these tests (Table 5) show the recrystallized material exhibited good ductility, particularly when deformed at 400°F, however the non-recrystallized material exhibited relatively poor ductility even after being annealed at 1800°F for 24 hours. Actual tube bending tests will be performed to determine the actual tube bending limits for this alloy.

Table 5
Results of Tube Flattening Tests

Tube Condition	Annealing Information		Hardness	Flattening % at Test Temperature	
	T (°F)	Time (h)	(HRC)	Ambient	400°F
CD / ANN	Standard HA Procedure		25	37.6	54.0
CD	N.A.	N.A.	35	0 (cracked)	0 (cracked)
CD/ANN	1700	1	26-28	5.9	6.5
CD/ANN	1700	24	26-28	4.4	8.2
CD/ANN	1800	1	26-28	4.0	5.3
CD/ANN	1800	24	25	6.2	9.0

Task 5: High Temperature Corrosion Limits of MA956
Laboratory Testing for Working Fluid Side: Work continues on the lifetime prediction of the MA956 alloy in air at very high temperatures. Testing at 1300°C has been completed and testing at 1200 and 1250°C is currently being performed. Figure 3 below shows a plot of a test conducted at 1300°C for the MA956 and MA956HT alloys.

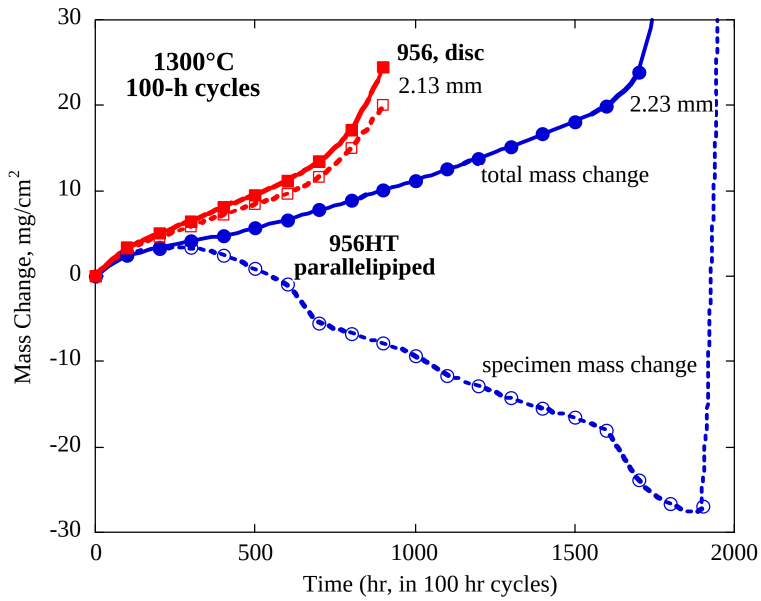


Figure 5. Plot of total mass change (solid line) and specimen mass change (dotted line) for the MA956 and MA956HT alloys oxidized in air at 1300°C.

Laboratory Testing for Fireside Environment: Laboratory testing using two different flue gases and three different deposits has been initiated. The composition of the flue gases and deposits are shown in Tables 4 and 5 below. A char analysis produced by the FWDC partial gasification unit was used as the basis for the deposit compositions.

Table 4
Flue Gas Compositions to be Used in Laboratory Fireside Testing

Species	Amount (vol %)	
	Gas Mixture 1	**Gas Mixture 2**
O_2	4	2
CO_2	15	15
H_2O	10	5
SO_2	0.25	1.0
N_2	Bal	Bal

Table 5
Deposit Compositions to be Used in Laboratory Fireside Testing

Species	Amount (wt%)		
	Ash 1	**Ash 2**	**Ash 3**
Si Dioxide	14.6	11.6	7.6
Al Dioxide	6.0	6.0	6.0
Ti Dioxide	0.3	0.3	0.3
Fe Oxide	1.3	1.3	1.3
Ca Oxide	3.3	3.3	3.3
Mg Oxide	0.3	0.3	0.3
Na Oxide	0.4	1.4	2.4
K Oxide	0.3	1.3	2.3
S Trioxide	1.2	2.2	3.2
P Pentoxide	0.3	0.3	0.3
KCl			1.0
Carbon	72.1	72.1	72.1

Field Exposure Testing: The location for field probe has been determined, the probe control hardware is currently being procured and the probe design is being finalized.

CONCLUSIONS

No technical conclusions are available at this time, however the change in grain morphology as a function of the extrusion + TMP parameters for the MA956 rods, and the joining obtained results thus far, are encouraging. Work will continue under Tasks 2, 3, 4, and 5 during the next fiscal year.

REFERENCES

1. Campbell, J., Jr., and Lee, J. C., "Fired Heater Versus CCGT/Cogeneration Cycle Parameters," ASME Paper No. 82-GT-187 (1982).
2. Klara, J. M., "HIPPS: beyond state-of-the-art, Part I," *Power Engineering*, 12, 37-39 (1993).
3. Klara, J. M., "HIPPS can compete with conventional PC systems: Part II," *Power Engineering*, 13, 20-23 Jan (1994).
4. Robson, F. L., Ruby, J., and Seery, D. J., "Repowering with High-Performance Power Plant Systems (HIPPS)," pp. 162-167 in *Proc. Pittsburgh Coal Conf.*, Sep. 1996.
5. LaHaye, P. G., Strom-Olsen, J., Seger, J. L., and Pickup, H., "Externally-Fired Combined Cycle Demonstration", in *Proc. Advanced Coal-Fired Power Systems '95 Review Meeting*, H.M. McDaniel, D.J. Mollot, and V.K. Venkataraman, Eds., DOE/METC-95/1018, pp. 55-64, June 1995 (1995)