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UTSI-98-03

**TECHNICAL PROGRESS REPORT**  
**FOR**  
**UTSI/CFFF MHD PROGRAM COMPLETION**  
**AND RELATED ACTIVITY**

**For The Period**  
**April 1, 1998 - June 30, 1998**

**July 1998**

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**Prepared for:**  
**The United States Department of Energy**

**Prepared by:**  
**The University of Tennessee**  
**Space Institute**  
**Energy Conversion Research and Development Programs**

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## **EXECUTIVE SUMMARY**

Routine preventive maintenance of the DOE Coal Fired Flow Facility (CFFF) is being performed. Modernization programs, funded under subcontract from Foster Wheeler Development Corporation by the DOE HIPPS Program, have been completed on the coal processing system, the data acquisition and control system and the control room.

Environmental restoration actions continued with monitoring of groundwater wells and holding pond effluent. Progress is reported on the approved groundwater remediation plan. Actions are underway to dispose of spent seed/ash mixtures and excess coal remaining from the MHD POC program.

The status of five (5) High Temperature Superconductor (HTS) projects is reported.

## **TASK 1 - FACILITY AND PROPERTY MANAGEMENT**

Activities this quarter were concentrated on the facility preventive maintenance/repairs required to maintain the Coal Fired Flow Facility in a standby condition. Weekly and monthly equipment maintenance procedures were performed for facility air compressor systems, cooling water pumps, coal processing system motors, steam boiler systems, ID and FD fans, and fire water system equipment.

The crankshaft of one of the high pressure natural gas compressors failed during a Westinghouse combustor test last year. The manufacturer, Ingersoll-Rand, completed repairs under warranty and returned the compressor to CFFF.

The radiant furnace was partially disassembled in anticipation of a new furnace for the discontinued HIPPS program. A plan to reconfigure the radiant furnace and portions of the superheater for operation was completed this quarter and reassembly work started. The disconnected downcomers were welded to the primary furnace and all open radiant furnace access ports were recapped. The rotary valves underneath the superheater were removed and blind flanges were installed at the bottom of the superheater hoppers.

Design work was initiated to complete the integration of coal system instrumentation and controls into the new Distributed Control System. This reconfiguration will provide for a more efficient and reliable coal system operation. Installation of coal system control valves, pressure regulators, and limit switches started.

The power monitoring instrumentation at the CFFF substation previously was not integrated into the control and data acquisition system. The software to access the data from the instrument, and the data logging and display was handled by a standalone PC. This arrangement is no longer usable, as it has experienced low reliability due to periodic lightning damage, and, the PC equipment was very old and obsolete. In addition, the data was not available in the new, upgraded control system. Work started to remedy this situation. Materials have been purchased and fabrication of a new interface panel is completed. This panel will interface the remote power instrument at the substation to the distributed control system (DCS). A new, optically isolated, RS-485 converter is included, as well as a fiber optic link to isolate the DCS from electrical transients on the communications lines due to lightning. A software driver for the instrument is being developed to bring the power data into the Factory link software for display and archiving.

Annual electrical substation and transformer maintenance was completed this quarter.

## **TASK 2 - REPORTING AND ARCHIVING**

All major pieces of equipment, mechanical and utility systems, and test hardware items in the CFFF have been documented with drawings and specifications. All hardware and systems added to the CFFF or modified since 1985 have been documented in AutoCAD format. Original drawings, microfilm copies and computer disks are maintained for archival purposes as well as daily use in maintaining the CFFF. The Design Engineering Department

continues to support the collection, filing and updating of CFFF mechanical system design documents.

### **TASK 3 - SITE ENVIRONMENTAL COMPLIANCE AND REMEDIATION**

Disposition of the remaining TRW tote bins has not been resolved.

Monthly water discharge monitoring and reporting has been completed on schedule. No violations were noted this quarter.

Table 1 shows the complete set of groundwater sample results. The two wells adjacent to the old septic field lines used by the Energy Conversion Programs showed some organic contamination. The contamination is local since chemical analyses of the water in wells downstream have shown levels below detection limits.

A letter will be sent to the State proposing a sample routine for three more quarterly samples. If these demonstrate similar results the State will most likely propose a "No Action" enforcement scheme.

Please refer to the May Monthly Report for the soil diesel excavation status. Due to excessive rainfall for July, this has hampered diesel evaporation efforts. However, the improvement is obvious from the much lesser odor. At the point where no odor is detectable, grab samples will be collected and sent for analysis. As stated in the May 1998 Monthly, no migration into the groundwater, as verified by lab analysis has occurred.

An audit by a Tennessee Air Pollution Control representative, Mr. Don Davis was performed. No discrepancies were noted. Mr. Davis did request and was given logs of testing that took place at the DOE CFFF, and total fuel usage.

Prepared and sent the UTSI Annual Safety and Health Report to the UT system office. This report includes the safety and health activities at the CFFF.

Table 1. Results of Chemical Analysis for Constituents of Concern

	UTSI-2	UTSI-3	UTSI-4	UTSI-9	UTSI-10	UTSI-11	UTSI-13	UTSI-14	UTSI-15	UTSI-16
Carbon Tetrachloride				BDL	BDL	44 ppb	BDL	BDL	BDL	BDL
1,1-Dichloroethylene		30 ppb		BDL	BDL	BDL	BDL	BDL	BDL	BDL
Tetrachloroethylene				BDL	42 ppb	88 PPB	BDL	BDL	BDL	BDL
1,1,1-Trichloroethane		7.7 ppb		BDL	BDL	BDL	BDL	BDL	BDL	BDL
Phenols	41 ppb			BDL	BDL	BDL	BDL	BDL	BDL	62 ppb
Chromium			15 ppb	25 ppb	59ppb	BDL	19 ppb	BDL	17 ppb	27 ppb
Copper	140 ppb		24 ppb	17 ppb	BDL	BDL	BDL	BDL	BDL	14 ppb
Lead				7.9 ppb	16 ppb	BDL	9.4 ppb	13 ppb	5.6 ppb	15 ppb
Nickel			110 ppb	23 ppb	70 ppb	BDL	39 ppb	41 ppb	19 ppb	110 ppb
Zinc	98 ppb		130 ppb	42 ppb	140 ppb	17 ppb	70 ppb	57 ppb	28 ppb	100 ppb
Diesel Residual Organics - Well #8 - Below Detection Level										
BDL = Below Detection Level										

#### **TASK 4 - SITE REACTIVATION**

No work was scheduled or performed.

#### **TASK 5 - DISASSEMBLY AND DISMANTLEMENT (D&D) OF THE CFFF**

No work was scheduled or performed.

#### **TASK 6 - ADVANCED TECHNOLOGY, RESEARCH, DEVELOPMENT AND ENGINEERING FOR OTHER FEDERAL OR DOE PROGRAMS**

##### **Subtask 6.02 Evaluation of Methods for Application of Epitaxial Layers of Superconductor and Buffer Layers**

During the reporting period of April 1, 1998 to June 30, 1998, the following progress was made in the above project.

The revised version of the topical report describing UTSLs evaluation of the candidate options has been submitted to Dr. Dean Peterson (LANL), Dr. Balu Balchandran (ANL), and Mr. Robert Hawsey (ORNL) for their comments. As of today, (LANL) and (ANL) comments have been received. As soon as we hear from the (ORNL), all the comments will be incorporated and the report will be distributed. The technical paper that was revised based on this report has been submitted to the Journal of Applied Superconductivity for possible publication.

From the two technical abstracts that were submitted for possible presentation at the upcoming ASC-98 conference to be held in Palm Desert, California, only one has been accepted for the oral presentation. We have not heard about the second abstract yet. The full manuscript describing the UTSL/ORNLs joint work on the bench scale dip-coating study will be prepared as soon as the graduate student's M. S. thesis is completed. Another abstract based on this bench scale work has also been accepted for presentation at the Annual AIChE Meeting to be held in November 1998 in Miami Beach, Florida.

The bench scale dip-coating unit and the glassware to prepare precursor solution/gel at UTSL are in place now. The single crystals of SrTiO<sub>3</sub> have been ordered. As soon as we receive them, the UTSL Chemist will coat them using precursor solution/gel of LaAlO<sub>3</sub>.

Efforts were made (at ORNL) in applying epitaxial YBCO layers over single crystals of SrTiO<sub>3</sub> (STO) and LaAlO<sub>3</sub> (LAO). Precursor solutions for these efforts included hexanoates, instead of alkoxides used in the sol-gel route. As a result, the chemistry was slightly different and can be considered as following the MOD route. Since the solution was quite viscous, the dip-coating method could not be used to coat the single crystal substrates. Epitaxial growth was achieved in YBCO/STO and YBCO/LAO samples with T<sub>c</sub> around 87K and J<sub>c</sub> measurement on one sample (measured by ORNL) equal to 1.83x10<sup>5</sup> amp/cm<sup>2</sup>. Epitaxial growth on the YSZ single crystals was not achieved and the reasons are being looked into.

Selected samples of rolled nickel substrates submitted by EURUS and annealed at UTSI were coated with  $\text{LaAlO}_3$  using UTSI's bench scale dip-coating unit and following the sol-gel route. From the analysis of about half of the samples, FWHM values for in-plane and out-of-plane orientations are found to be around  $8-10^\circ$  and the epitaxy of about 50 percent seems to have been achieved. Since the starting nickel samples also had the FWHM values ranging from  $8-12^\circ$ , it seems that unless the texture of the starting metallic substrate is improved further, a good epitaxial growth by the sol-gel route over rolled metal, may not be possible. Some of these  $\text{LaAlO}_3$  coated nickel samples have been given back to EURUS for applying YBCO coating by e-beam and/or liquid phase epitaxy (LPE) methods.

A plan for the next fiscal year work at UTSI has been put together that includes continuing the joint effort with ORNL in further developing the solution growth based YBCO application techniques. As soon as the new graduate research assistant designated for this work starts at UTSI in the fall of 1998, this work will be initiated. The current GRA, who is finishing his M.S. degree, is analyzing the experimental data and putting together a detailed description of the work done, so far, at UTSI/ORNL in the area of sol-gel based application of buffer and HTS material. This will be reported in his M.S. thesis, which will be later modified as necessary, for distribution as a topical report.

### **Subtask 6.03 Coated Conductor Development and Program Management**

The primary activities under this task have been consultation with members of the advisory committee, and others, on the need for any modifications in the Roadmap for Coated Conductors. Preparations were started for presentations to the DOE Peer Review in July.

### **Subtask 6.04 Optimum Coated Conductor**

As reported in the May monthly progress report, the discovery of aluminum contamination on one side of all the previous samples annealed and examined by AFM has required that all samples be prepared again using an improved annealing procedure. This has been completed and the following measurements are un

- Atomic Force Microscopy
  - Groove Dimensions
  - Dihedral Angle

- X-Ray Diffraction
  - Overall Texture

- Backscattered Kikuchi Diffraction
  - Misorientation of individual grains

- Optical Microscopy
  - Grain Size

By the end of the quarter, these measurements were nearing completion and a report is being prepared on this work.

**Subtask 6.05 Cost/ Performance Analyses of Potential Manufacturing Processes**

As reported in the June Monthly Contract Status Report, the draft report on the economic comparison of the electron beam and pulsed laser deposition was revised to final draft status and forwarded to interested industrial participants for their review and recommendations. One revision included calculating the effects of different thickness of superconductor. The base case considered in the study was for a superconductor thickness of 2 microns. With this superconductor thickness, it is not possible to meet the fill factor goal of 10% (superconductor cross section/total wire cross section). Table 2 shows the results of this analysis.

**Table 2**  
**COST EFFECT OF SUPERCONDUCTOR THICKNESS**  
 ( $J_c=10^5$  a/cm<sup>2</sup> CASE)

T(m)	FF(%)	<u>Electron Beam Process(\$)</u>		<u>Pulsed Laser Deposition(\$)</u>		
		I <sub>c</sub> (a)	Cost/m	Cost/kA-m	Cost/m	Cost/kA-m
2	4.3	40	1.59	39.90	3.01	75.20
3	6.3	60	1.75	29.17	3.88	64.70
4	8.3	80	1.90	23.81	4.75	59.44
5	10.2	100	2.11	21.06	5.63	56.28
6	11.9	120	2.28	18.97	6.50	54.18
7	13.7	140	2.44	17.47	7.37	52.68
8	15.3	160	2.62	16.35	8.25	51.55
9	17.2	180	2.79	15.48	9.12	50.67
10	19.2	200	2.96	14.79	9.99	49.97

**Subtask 6.06 Development of Real Time Process Control Using InSitu Diagnostics**

Raman Spectroscopy

Work has continued in the Raman spectroscopy lab extracting Raman data from numerous samples. Raman data has been taken on a YBCO film sample, a lanthanum aluminate film sample, and single crystal materials of buffer layers.

YBCO Raman scans were performed using higher laser powers than originally used, 50-60 mW incident power. These scans were performed using several different polarization dependencies. No significant Raman data was obtained from this film sample. In two scans the strongest Raman line for a (001)-oriented film at 335 cm<sup>-1</sup>

seemed to appear, but was extremely broad. A broad Raman line would suggest that the film does not have a well-ordered and homogeneous crystal orientation. The data collected from these scans suggest that either the film sample has small, unordered grains or a flaw exists in the experimental setup. The experimental setup used is a typical double spectrometer/photomultiplier tube backscatter configuration used by many groups to obtain Raman data from YBCO samples.

Also this setup has provided Raman data from other samples such as diamond and sulphur. Improvements may need to be made to the setup to focus the incident laser light into a smaller region on the sample, use a photomultiplier tube with a lower dark noise, or use a triple spectrometer setup with a higher rejection ratio than the double spectrometer setup. Such improvements may allow the setup to obtain a higher signal to noise ratio, but may not necessarily provide significantly better data on this specific YBCO film, since it appears to be a low quality film.

Raman spectra have also been taken from lanthanum aluminate film samples. These samples showed three strong Raman or groups of Raman lines. Two lines were found from the lanthanum aluminate layer itself and another line was found from the substrate, which is strontium titanate. For thicker layers of lanthanum aluminate the Raman lines from that layer were stronger than the line from the substrate. A possible technique using these Raman lines could be developed to determine the layer thickness of the lanthanum aluminate layers. This technique would be similar to that proposed to be used for the HTSC film layers.

Raman work has also proceeded on some buffer layer single crystal samples. These samples are cerium oxide, magnesium oxide, yttrium stabilized zirconia, and lanthanum aluminate. These samples are being investigated for Raman lines. Work has been slow due to the lack of air conditioning in the Raman spectroscopy laboratory caused by the electrical power modernization construction on campus.

### Atomic Absorption Spectroscopy

An experimental apparatus was designed and partially constructed to determine the feasibility of using atomic absorption spectroscopy as a control measurement of gas flow concentrations of YBCO precursors in the HTSC Metalorganic Chemical Vapor Deposition (MOCVD) processing scheme. The experimental configuration consists of a deuterium lamp, collimation optics, a high temperature oven, a vaporization chamber, a precursor sample tray, argon purge plumbing and vacuum system, focusing optics, and a spectrometer. Spectral data acquisition will use a PC based LabViewJ program which is being modified for these tests.

The Y, Ba, and Cu precursor vaporization chamber was obtained with sapphire windows that transmit UV, visible and near IR light and appropriate seals for a 1 Torr vacuum. Identical oven windows were obtained also, with supracil™ lenses and mirrors selected with equal transmission characteristics to provide a broad wavelength regime for absorption peak determination. The precursor sample tray was machined to mount in the vaporization chamber and glove bags were obtained to allow the precursor sample transfer without exposure to CO<sub>2</sub> or water vapor. While waiting for the chamber to arrive a mock up was built to test the vacuum pump capabilities and also to develop procedures to move measured masses of precursors from the inert glove box storage to the chamber. The vacuum pump was obtained and tested, and chamber pressures at 1 to 10 Torr were established with adequate pumping time. Mounting hardware was built for the deuterium lamp, the oven, and the spectrometer, and the optical benches,

lamp, mirror, apertures and spectrometer were aligned. The vaporization oven was modified with windows and chamber mounting hardware, and quick disconnects to the vacuum-purge system. However delays have occurred because the experimental chamber had to be modified to meet specifications and a clerical error delayed the arrival of the lenses. But the modified chamber and critical focusing lens are scheduled to arrive early next quarter and the first trial runs are planned with the completed experimental setup within the first month of next

Objectives for next quarter include vacuum testing the vaporization chamber at temperature, finalizing procedures to load the precursor samples in the chamber, assembling the rest of the optical system and the vacuum system, to complete the hardware setup. Additionally, the LabView™ software for data acquisition will be modified and data will be collected with known gas sources and then precursor samples in the UV regime.

#### Laboratory Support

X-ray diffraction analyses continued in support of Eurus Technologies, Inc., Westinghouse Electric Company, Midwest Superconductivity, Inc., and the UTSI/ORNL work in Sol-Gel coatings.

#### OPEN ITEMS

**A. UTSI:** None

**B. DOE/FETC:** Topical Report entitled, "Evaluation of Methods for Application of Epitaxial Buffer and Superconductor Layers" sent for final review and specification of distribution.

Property Retirement Work Order Authorization #15-31 awaiting process and approval.

#### SUMMARY STATUS ASSESSMENT AND FORECAST

The CFFF continues in a standby status with preventive maintenance and repairs being accomplished as funding permits. Utilization of the facility for other work, with DOE authorization, provides assistance in maintaining operational capability. Currently, tests of the Westinghouse Electric Corporation topping combustor are the only tests scheduled in the facility.

High Temperature Superconductor work continued substantially as scheduled on the five subtasks (6.02 - 6.06) previously authorized. Additional cooperative programs are being discussed.

The groundwater remediation at the CFFF is progressing in accordance with the approved plan.

### **TASK AND COST VARIANCES**

The variance in Tasks 1 - 3 continue to be positive because the available funding level is below the Management Plan level for these tasks.

The positive variance in Task 6 reflects management decision to reduce FY-98 expenditures below Management Plan levels.

**APRIL 1, 1998-JUNE 30, 1998 QUARTERLY VARIANCE REPORT**

Planned vs. Actual Expenditures

(thousands of dollars)

TASK	PLANNED	ACTUALS	VARIANCE
1	208.3	177.6	30.7
2	98.9	32.2	66.7
3	79.7	51.0	28.7
4	0.0	0.0	0.0
5	0.0	0.0	0.0
6	465.6	395.5	70.1
TOTALS	852.5	656.3	196.2
COST ELEMENT			
DIRECT LABOR	349.3	343.4	5.9
FRINGE BENEFITS	87.3	73.0	14.3
EQUIPMENT	119.1	17.0	102.1
EXPENDABLE MATERIAL	34.8	8.8	26.0
OUTSIDE CONTRACTS	10.2	6.5	3.7
TRAVEL	7.3	1.8	5.5
TOTAL DIRECT COSTS	608.0	450.5	157.5
INDIRECT COSTS	244.5	205.8	38.7
TOTAL	852.5	656.3	196.2

Planned vs. Authorized Funding

Cumulative

Task	PLANNED	AUTHORIZED FUNDING
1	2004.6	
2	1180.7	
3	923.3	
4	153.0	
5	118.0	
SUBTOTAL	4379.6	1857.7
6	4212.0	3351.3
TOTAL	8591.6	5209.0