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TECHNICAL PROGRESS REPORT
FOR
"HIGH PERFORMANCE MATERIALS IN COAL CONVERSION UTILIZATION"

FOR THE PERIOD
JANUARY 1, 1994 - MARCH 31, 1994

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Table of Contents

	<u>Page</u>
1.0 <u>INTRODUCTION</u>	1
2.0 <u>DISCUSSION</u>	1
2.1 Objective and Scope of Work	1
2.2 Task 1- Materials (Lanxide Corporation)	1
2.3 Task 2- Pre and Post Test Material Characterization	2
2.3.1 Subtask 2A- Strength of Materials Testing and Analysis (UTSI)	2
2.3.2 Subtask 2B Corrosion Thermodynamic Analysis (U of Pa)	2
2.4 Task 3 Exposure Testing	3
2.4.1 Subtask 3A Bench Scale Lab Tests (UTSI)	3
2.4.2 Subtask 3B Field Exposure Tests (UTSI)	5
2.5 Task 4 Project Management	5
3.0 <u>SCHEDULE</u>	5
4.0 <u>CONCLUSION</u>	6

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List of Figures

Page

Figure 1. Slag Dispersal System for Bench Scale Exposure Testing	4
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1.0 INTRODUCTION

This is the second quarterly report concerning this three year grant on "High Performance Materials in Coal Conversion Utilization." The grant is for a joint university/industry effort under the US Department of Energy (DOE) University Coal Research Program. The University of Tennessee Space Institute (UTSI) is the prime contractor and The University of Pennsylvania and Lanxide Corporation are subcontractors.

The administrative details involved in the finalizing of the subcontracts and the acquisition of the cost shared equipment by UTSI and the U of Pa has slowed progress somewhat, however, the project should go smoothly from this point on. Most of the efforts of this quarter have gone toward preparing the equipment and plans for the laboratory tests. Research into the identification of a suitable protective coating based on thermodynamic considerations and the development of techniques for their application as well as joining ceramic materials has been initiated.

2.0 DISCUSSION

2.1 Objective and Scope of Work

The object of this grant is to test, analyze, and improve the heat and coal-slag corrosion resistance of a $\text{SiC}_{(p)}/\text{Al}_2\text{O}_3$ ceramic composite tubular material. The material will be evaluated for its ability to withstand the pressures, temperatures and corrosion attack which will be encountered within a coal-fired high-temperature, high pressure air heater. The evaluation will include strength testing at elevated temperatures. Joining and coating techniques will also be investigated.

2.2 Task 1- Materials (Lanxide Corporation)

Under Lanxide's cost share portion of the grant, they will provide 21 183 cm (six foot), 5 cm (two inch) OD $\text{SiC}_{(p)}/\text{Al}_2\text{O}_3$ DIMOX™ ceramic composite tubes for testing by UTSI. Lanxide provided a shipment of 5 tubes (materials designation #93-X3015) during 1993 and 10 tubes (material designation #93-X3063) so far in 1994. Both of these deliveries were standard production tubes.

The remaining six tubes will be provided to UTSI later in the year. These tubes will be used to determine if different manufacturing processes can be used to improve quality and reduce manufacturing costs. The tubes will be in three groups of two each and will be processed as follows:

Group 1- Lower cost SiC powders will be evaluated. A slip system will be evaluated which has the potential of reducing material costs by at least 40%. The material will be evaluated by lab tests and compared to the current commercial material test results.

Group 2- Extrusion will be evaluated as a low cost alternative to slip casting for preforming tubes. This again will be tested and compared to the results from the commercial tubes.

Group 3- Demonstrate scale-up of the "growth" process used to produce the alumina matrix in the composite. In addition, the effectiveness of a new high density furnace will be established. This again will be tested and compared to the results from the commercial tubes.

In addition to the foregoing, Lanxide will refine their quality assurance program to develop procedures, guidelines and documentation for proof testing of tubes as a QC tool for the final product. They will also develop a set of process control methodologies that track the product through preforming, growth, and post growth processing steps. These controls will be based on statistical processing approaches. They will address dimensional as well as microstructural quality. The procedure will be documented via a standard operation procedure.

2.3 Task 2- Pre and Post Test Material Characterization

2.3.1 Subtask 2A- Strength of Materials Testing and Analysis (UTSI)

UTSI's cost share contribution to this grant consists of upgrading the UTSI MTS Systems Corporation testing machine for the testing of ceramics at elevated temperatures. The procurement was bid and issued to MTS Systems Corporation. The installation of the new high temperature (1600°C max) furnace and ceramic extension rods for C-ring compression tests will be completed during the next quarter. Once the installation is complete, the tubes that were exposed during the coal-fired MHD test will be sectioned and tested in the MTS apparatus.

Based on the constraints associated with constructing ceramic grips for mechanical tension testing of O-ring sections along with the more reliable prediction of the stress distribution in C-ring sections, it has been concluded that the best way to conduct tension tests in the MTS setup will be to compress C-ring sections. A computer code has been developed which determines the stresses associated with this test configuration as well as the appropriate Weibull parameters.

2.3.2 Subtask 2B Corrosion Thermodynamic Analysis (U of Pa)

The preliminary evaluation of various software packages for thermodynamic modeling was continued until the subcontract was signed and funding was received at the later part of the quarter. After comparing demonstration versions and literature information, the program, MTData (from AEA Technologies) was found most suitable for this project. This software is basically a Gibbs Energy minimizer (with a database), for calculating multicomponent phase equilibria. Hardware on which to run the program is currently being evaluated and will be purchased as soon as possible.

2.4 Task 3 Exposure Testing

2.4.1 Subtask 3A Bench Scale Lab Tests (UTSI)

Two UTSI furnaces have been equipped with temperature controllers and are presently ready for the test series. One will be used to heat clean tubes and the other will be for the slag exposed tubes. The following temperatures will be used for each 200 hour test on both the cleaned and exposed tubes:

1100°C (2012°F)
1260°C (2498°F)
1400°C (2696°F)

The exposed tube furnace has been modified to accommodate a video camera and a lapsed-time video taping will document the slag deposits on the test tube. An automated slag dispersal system was added to the furnace so that a fresh layer of ground coal slag can be sprinkled on the tube every hour. (See Figure 1).

About 10 liters of slag produced from Illinois #6 coal was provided by Southern Illinois Power. A slag sample was re-ashed by UTSI in March 1994, and analysis was performed with the following normalized results:

Water	2.25 %
Ash	97.75
Elemental (assumed oxide)	
Al ₂ O ₃	16.57 %
CaO	10.37
Fe ₂ O ₃	19.79
K ₂ O	1.48
MgO	2.62
Na ₂ O	0.37
SiO ₂	47.76
TiO ₂	0.85
SO ₃	0.19
Total	100.00 %

The remaining slag is being pulverized to less than 250 μm in preparation for the exposure testing.

An investigation into protective coatings has been undertaken by UTSI. An aluminum coating will be applied to a Lanxide SiC_(p)/Al₂O₃ composite via physical vapor deposition. This will then oxidized by heat treated to form alumina. A laser will then be used to surface treat the coating to enhance the bonding between the surface coating and the composite substrate. The effect of various wavelengths (CO₂= 10.6 μm , Nd-YAG= 1.06 μm and excimer= 248 nm) and modes (pulse or continuous) of operation on the bonding characteristics will be evaluated. The microstructure of the coating interface will be studied by SEM, EDS, and X-ray mapping. The

FIGURE 1.
 Slag Dispersal System for Bench Scale Exposure Testing

adherence of the coating will be evaluated and if warranted the samples will be exposed to heated slag and strength tested.

Preliminary attempts to develop a concept and establish a general procedure for laser joining of monolithic SiC ceramics and SiC_(f)/SiC matrix and C_(f)/SiC matrix ceramic composites have been conducted with reasonable success. The ceramic materials have been joined with a titanium-base interface reactant material. The joints thus created are believed to possess properties compatible to those of the base ceramic materials. The joining was conducted in air with high laser traverse speeds ranging from 1500 mm/min to 3500 mm/min using a pulse width modulated (PWM) CO₂ laser (10.6 μm) beam at suitable power levels (350-700 watts). Based on these initial trials, it appears that the laser joining techniques for ceramics holds great promise and can be developed further for a variety of ceramic materials.

2.4.2 Subtask 3B Field Exposure Tests (UTSI)

The termination of the MHD program has limited field testing to the five tubes exposed during the final MHD test. At the request of the DOE Contract Monitor, Cliff Smith, one of these tubes was delivered to DOE for additional exposure tests at a different location. The tube provided was tested at UTSI for 52 hours at 1038°C (1900°F) which was within 53°C of one of the other tubes and thus redundant to UTSI's needs. This leaves the following tubes for testing in the MTS furnace/machine:

<u>Lanxide Ceramic Composite Tubes- Delivery #1</u>			
<u>52 Hours in Potassium Seeded Montana Rosebud Coal Exhaust</u>			
Tube 1.	1399°C	(2550°F)	Reducing Zone
Tube 2.	1260°C	(2300°F)	Reducing Zone
Tube 3.	1093°C	(2000°F)	Reducing Zone
Tube 4.	1149°C	(2100°F)	Oxidizing Zone

2.5 Task 4 Project Management

Mr. Bill Boss will attend the Eighth Annual Conference of Fossil Energy Materials in Oak Ridge in May 1994 and the Tenth Annual Coal Preparation, Utilization, and Environmental Contractors Conference in Pittsburgh in July 1994.

3.0 SCHEDULE

Lanxide has delivered 15 tubes, five of which have been exposed to potassium seeded coal slag for 52 hours. The others will be exposed shortly in the laboratory furnaces. The MTS machine upgrade is taking longer than anticipated, however, there is sufficient work to be done prior to its operation so that this not a significant problem.

4.0 CONCLUSION

Good progress has been achieved toward the objectives of this grant. The second set (10) of Lanxide tubes will soon be exposed to the laboratory series of tests. At the completion of these tests, the MTS mechanical testing machine should be ready for the strength tests. Significant progress has been made in the areas of coating and joining while awaiting the results of the mechanical testing.

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