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

For The Period
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Prepared for:

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Prepared by:

The University of Tennessee Space Institute
B. H. Goethert Parkway
Tullahoma, Tennessee 37388-8897

MASTER

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1.0 INTRODUCTION

This is the first quarterly report for this three year grant on "High Performance Materials in Coal Conversion Utilization." The grant is a joint university/industry effort under the 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.

It was initially planned to field test ceramic composite tubes furnished by Lanxide Corporation in conjunction with an on-going DOE magnetohydrodynamic (MHD) test series at UTSI. The MHD test program was curtailed due to funding limitations near the beginning of the grant so that the field test portion is now greatly reduced. Bench scale testing will replace most of the field testing. This development should have minimal effect on this research since there is now little interest in the affects of the potassium seeded MHD coal ash on heat exchanger surfaces.

2.0 DISCUSSION

2.1 Objective and Scope of Work

Test and analyze the heat and corrosion resistance of a $\text{SiC(p)/Al}_2\text{O}_3$ 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.

2.2 Task 1 Materials (Lanxide Corp.)

Lanxide provided five each 183 cm (six feet), 5 cm (two inches) OD SiC(p)/Al₂O₃ composite ceramic tubes for testing. These deliveries were part of Lanxide's cost sharing contribution to this grant. The final tube deliveries will be early in 1994.

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 contribution to this grant consists of upgrading the UTSI MTS Systems Corporation testing machine for the testing of ceramics at elevated temperatures. The upgradation includes installation of high temperature furnace ($\approx 1600^{\circ}\text{C}$) and high temperature material grips. This procurement was begun at the end of the quarter and the contract award should occur in February 1994.

A theoretical study was performed to determine the location of maximum tensile stresses in an O-ring under various uniaxial loading conditions. It was found that these maximum tensile stresses would be on about one-fourth of each inside surface facing diametrically opposite to each other along the loading axis of the ring if loaded in compression, and on the outside surface if loaded in tension. Tension loading will allow the location of the critical stresses to coincide with the location of the coal slag deposits on the external surface of the exposed tubes. The O-ring geometry is chosen based on the consideration that the stress distribution is symmetric and more predictable which in turn eliminates the effect of stress concentration on the strength of the material associated with C-ring geometry.

A FORTRAN code is currently being developed which will use the stress distribution equations developed in this study to determine the statistical parameters necessary to predict the failure of the test materials.

Further discussions with Lanxide Corp. and Oak Ridge National Labs (ORNL) are currently underway regarding setting up standard procedures for mechanical testing based on their experience in this area.

UTSI has been developing a procedure of analytical analysis for studying structure-property relationships of ceramic material used in the present efforts. The procedure includes techniques such as SEM, Electron-beam Microprobe Analysis and Qualitative/Quantitative Image Analysis. The actual analysis will begin after the MTS machine is upgraded and the samples have been exposed.

UTSI team visited ORNL on January 26, 1994, and has conducted preliminary discussions concerning testing and analysis techniques of structural ceramics.

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

Although the subcontract with the University of Pennsylvania has not been finalized, the University has conducted a survey of appropriate codes for their thermodynamic analysis. This code will be their cost contribution to the grant.

2.4 Task 3 Exposure Testing

2.4.1 Subtask 3A Bench Scale Lab Tests (UTSI)

Two furnaces acquired from DOE Excess Property are being modified for the bench scale tests. A work order was issued to provide controllers for the furnaces to replace present thermostats. An automated ash dispersal system is being developed

to permit the deposits on the tube undergoing testing or exposure to be refreshed about every hour.

A video system will be used to document the ash dispersal system. The video will be time compressed to conserve tape.

An investigation has been initiated into techniques for measuring the gaseous environment within the furnace. The actual gases to be measured have not been finalized; however, oxygen will be included in the selection.

2.4.2 Subtask 3B Field Exposure Tests (UTSI)

The five tubes furnished by Lanxide under Task 1 were exposed during the last MHD test in the MHD program. A 30 cm long section from the closed end of each of the five tubes supplied by Lanxide was cut off. Five 152 cm (five feet) lengths of the Lanxide tubes were exposed during the MHD test (LMF5-J) in the DOE Coal Fired Flow Facility (CFFF) radiant furnace. During the 52-hour long test, four tubes were exposed to the reducing environment of exhaust gases from Montana Rosebud coal seeded with a potassium compound, while one tube was located further downstream in an oxidizing environment. The respective tube temperatures were:

1399°C (2550°F),
1260°C (2300°F),
1093°C (2000°F),
1038°C (1900°F),
and 1149°C (2100°F).

These tubes will be sectioned and tested in the MTS machine after completion of its upgrade.

Two of the 30 cm closed-end tube sections were used to test a compression technique for sealing the tubes. One was pressurized to 14.4 atm. and held for an hour. The tip of the tube was around 1482°C (2700°F) while the seal area was measured at around 371°C (700°F). The other was similar but at a lower temperature. It was discovered that there were several very small leaks in the plumbing that normally would be undetectable in an open system. After the test, the apparatus was submerged in water while pressurized to 7 atm. All the leaks disappeared except for very small cobweb-like air streams coming from the seal and from the closed end of the tube via a pore. Work on this and other sealing techniques will continue as time permits during the rest of the research.

2.5 Task 4 Project Management

The PI for this grant, Dr. T. Dwayne McCay, was selected to be the Vice President for The University of Tennessee Space Institute. He will continue as PI for the grant.

A project kick-off meeting was hosted by UTSI. In attendance were: UTSI (Dwayne McCay, Bill Boss, Naren Dahotre, Peter LaRue, and Xiao Chenghe), Lanxide (Will Johnson), the U of Pa (Wayne Worrell), ORNL representatives (Vic Tennery, Roddie Judkins and Kristin Breder) and the DOE technical contract monitor (Cliff Smith).

The test temperatures were discussed at the meeting and there was follow-up discussion via electronic mail. It was agreed to by all that the temperatures should be lower and should be the same as those used by ORNL and the University of North

Dakota Energy & Environmental Research Center (UNDEERC) so that comparisons can be made. The temperatures for the lab scale tests are:

1100°C ORNL & UNDEERC

1260°C UNDEERC

1400°C ORNL & UNDEERC

UTSI plans to periodically sprinkle the coal ash on one surface of a Lanxide tube while it is heated to each of the foregoing temperatures for 200 hours. The 200 hours exposure time was selected as a standard based on the duration of the proposed (and canceled) future MHD tests. It may be advantageous to also consider testing periods which correlate to the ORNL and UNDEER test times.

Since there are no more MHD tests scheduled, it was agreed that the coal ash corrosion could be simulated in the lab. It was decided that unseeded Illinois #6 coal ash would provide the best environment since so much work had been done with this ash and the future needs of an MHD air heater are distant (i.e., interest in a potassium seeded ash has diminished). The results of this study then could easily be compared to other similar efforts. Southern Illinois Power has offered to send about 14 kg of ash for UTSI's tests.

3.0 COST STATUS

The start up activities for this grant and administrative delays in the signing of the subcontracts have kept the costs below the budgeted amount. Next quarter will see the project costs approach the budgeted level.

4.0 SCHEDULE

The project got off to a good start with the field testing of five Lanxide tubes. Activity slowed while preparations were being made for the bench scale exposure and mechanical tests. Next quarter should see several bench exposure tests completed, some mechanical tests finished and a good start on the analytical analysis.

5.0 CONCLUSION

This grant has made satisfactory progress as the foundations for the research were initiated. Activities will increase next quarter after equipment is installed and procedures are developed. No problems are envisioned at this time.

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