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DIST. CATEGORY UC-112
UTSI-00-2

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

For The Period
April 1, 2000 – June 30, 2000

August 2000

Work Performed Under Contract No. DE-AC22-95PC95231--32

Prepared for:
The United States Department of Energy

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

DISCLAIMER

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EXECUTIVE SUMMARY

Maintenance work on the DOE CFFF facility and other related government property is no longer authorized under this contract in accordance with the DOE-UT Settlement Agreement.. Environmental remediation preservation of the facility continued. Government property has been transferred to UTSI as owner which frees up many items for proper disposal. Actions are underway to dispose of other wastes, and control pests and water at the DOE CFFF.

Only one high temperature superconductivity project is active under Task 6. Under the particular Subtask 6.02, samples of nickel obtained from Plastronics, Inc. were coated with the LAO precursor solution. These coated samples have been characterized using the x-ray diffraction unit, and the results from the x-ray diffraction characterizations were further analyzed using a statistical program to develop multi-regression coefficients. Based on these regression coefficients, the variables that have significant effect on the coating of LAO over nickel substrate have been identified. A Master of Science thesis was prepared describing these findings and it will be defended in front of the committee members during July, 2000.

TASK 1 – FACILITY MAINTENANCE AND PROPERTY MANAGEMENT

In September 1998, a stop-work order was issued for work activity in the DOE Facility maintenance area. Work to administer government-owned property continued.

On September 22, 1999, Department of Energy and the University of Tennessee signed a SETTLEMENT AGREEMENT resolving all claims and property accountable under Tasks 1-5.

TASK 2 - REPORTING AND ARCHIVING

April, May and June 2000 Monthly Reports were submitted.

The Quarterly Key Staffing Report for the period January – March, 2000 was submitted April 18, 2000.

Quarterly Technical Progress Report for the period January-March 2000, was submitted April 26, 2000.

TASK 3 - SITE ENVIRONMENTAL COMPLIANCE AND REMEDIATION

UTSI shall continue implementation and compliance with the State of Tennessee approved plan for groundwater remediation. UTSI shall remove from the site, and properly dispose of, all industrial type non-hazardous wastes. In addition, UTSI shall properly remove all asbestos-containing cooling water tower materials and dispose of these materials properly. In addition, UTSI shall continue the monitoring and treatment of holding pond effluent per the site Water Discharge Permit with the State of Tennessee.

- General property disposal/disposition is still under discussion. There are disposal issues associated with these discussions.
- A report was received in April that showed the results of the groundwater sampling events that took place in March. Wells 10 and 11 were approximately at the same levels that have been seen in years past. Conversations with the State of Tennessee enforcement Division indicate that the State may require a more aggressive approach to the VOC contamination associated with wells 10 and 11.
- UTSI Well #8 is located below the remediated diesel source. This well was sampled for EPH (test associated with petroleum spills) in March, and showed negative for EPH. The diesel source may be closed as a groundwater issue.

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, RESEARACH, DEVELOPMENT AND ENGINERING FOR OTHER FEDERAL OR DOE PROGRAMS

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

During April-June 2000, all the efforts were spent in documenting the results from our experimental work as well as from our process design work in a thesis-document form. There were two drafts of this thesis prepared and reviewed before it was submitted to the thesis committee for approval. The excerpts from the version that the thesis committee is looking at are as follows:

Major Conclusions with Regards to Experimental Results:

The effects of several processing parameters on epitaxial growth of $LaAlO_3$ on cube-textured nickel were successfully characterized using the TaguchiL12 screening experiments. The epitaxial growth was performed without annealing of nickel before coating. The characterization involved the development of a non-dimentional term, epitaxial number, to weigh together the in-plane and out-plane sharpness along with the composition of each crystal orientation. This characterization also involved the successful development of variance and response models that could be used to identify trends in the epitaxial number and to differentiate significant factors from the insignificant ones. These models were developed on the sole basis of each experimental test (phi, omega, and phase scans) results and then on the combined performance number (epitaxial number) to confirm the existence of trends in the responses and to locate the origins of them.

The experimental results suggested that the major factors in determining the epitaxial growth were the oven temperature, solution concentration, oven time, and oven atmosphere. Increasing oven time and temperature while decreasing solution concentration and maintaining 4% hydrogen in argon during thermal treatment, produced higher epitaxial numbers. Eight confirmation runs were used to validate the above trends. Results showed marked improvement in all aspects (phi FWHM, omega FWHM, phase ratio). The phase scans showed the presence of one dominant peak of $LaAlO_3$(100). The average FWHM of omega and phi scans were 11.94° and 12.31°, respectively. These were comparable to the omega and phi FWHM average values of 10.55° and 15.75° reported by Vineet Lasrado using the ORNL supplied RABiTS nickel that had been annealed prior to dip-coating (Lasrado 1998). In-plane twins were also found at half the intensity as the (100) oriented $LaAlO_3$. These were also reported in the earlier work done by Vineet Lasrado.

The processing parameters that produced the improved results were as follows:

Oven Temperature = 1150°	Oven time = 1 hour
Oven Atmosphere = 4% H_2 in Ar	Solution Concentration = 0.10M
Withdrawal Rate: = 3 cm/min	Degree of Hydrolysis = 1.3

Surface Roughness = 0 (Electropolished State)

Samples that were electropolished prior to buffer coating also had an identifiable trend of improved epitaxial growth. Optical profilometer scans of surface roughness of polished and unpolished scans were, however, inconclusive and did not show any improvement in the surface roughness with the electropolishing work done here. Instead, the improved results may have been linked to the etching away of possible surface contaminants during the electropolishing process. In addition, optical profilometry indicated that the surface roughness increased between unannealed and annealed cube-textured nickel samples. Ra values for surface roughness increased from 0.43 to 0.98 microns with annealing times in excess of twenty hours. This may be explained by thermal grain boundary grooves developing during the annealing process.

The use of an intermediate cube-textured NiO layer was explored for processing in air and improved textures. The results indicated that it provided neither. At temperatures above 1050°C, the NiO layers broke down. In air, the NiO peaks grew as shown by the phase scan, but the $LaAlO_3$ peaks disappeared. Some phase scans indicated an enlarging of the lattice parameters suggesting possible cross-contamination between the $LaAlO_3$ and NiO.

<u>Major Conclusions with Regard to Process Economics:</u>

A sol-gel manufacturing cost evaluation was carried out to determine the product cost ($/kA-m) and to compare it with the other production methods under the comparable production rates and specifications. The product cost for the base case conditions was found to be $5.65/kA-m. This is within the objective of $10/kA-m for economic feasibility for large-scale electrical applications. In comparison to other processing methods explored earlier by Dr. James Chapman of UTSI and Robert Hammond of Stanford University, the sol-gel based processing scheme has a product cost greater than e-beam with RABiTS substrate and very similar to MOCVD ($5.6/kA-m).

The sensitivities to several key assumptions including material utilization, YBCO thickness, critical current density, plant capacity, withdrawal velocity, and raw material prices were also examined. As capacity factor increased, the product cost decreased exponentially. Increasing the material costs led to linear growth in the product cost. By increasing the material utilization factor, the product cost decreased exponentially. Decreasing the critical current density led to a sharp exponential rise in the product cost. Decreases in the YBCO film thickness led to higher product costs. The following limits for each sensitivity variable were identified to remain within the product cost objective.

YBCO thickness > 1.75 microns $J_c > 5x10^5$ *amps/cm*^{2} *Material Utilization Factor > 0.30*

Plant Capacity Factor > 0.25 *Raw Material Price < $1750/lb*

The limit for withdrawal velocity was not determined, but the product cost was shown to rise exponentially to approximately $8/kA-m at withdrawal rate less than 10 cm/min.

Finally, the concept of having precursor solution not produced on site was also explored. For unit price of $168 per gallon for precursor solution the product cost would remain unchanged. Also, in order to meet the $10/kA-m goal, the precursor price should not exceed $440 per gallon.

Based on our work, the following recommendations are made regarding future work.

- In terms of future characterization of epitaxial growth, it is important to investigate interaction terms when modeling the coating process. Time and temperature during thermal treatment and coating operation may have some interactions. In addition, solution concentration may also form a three-way interaction with these terms due to the fact that higher solution concentrations probably mean more material that has to be crystallized in the same period of time and under similar thermal inputs. Also of interest might be investigations into porosity of the buffer and HTSC layers prior to thermal treatment. Porosity could account for increases in thermal treatment time due to increased diffusion times for atoms to migrate to the growing expitaxial grains. They may also act as possible dislocation sites where the orientation of crystals forming around may be altered in orientation.

- In order to keep the sol-gel product cost within the goal, the most influential factors (labor and material costs) must be addressed. Savings in labor can result through further automation or reduction in the number of manual tasks needed. Adjustments to the width and length of processing tape dimensions should be considered to increase the amount of material on a spool. This would increase the ratio of process time to transition time for processes like dip-coating and batch thermal treatment for subsequent crystallization. Consequently, there will be a reduction in labor through less time and less number of sessions of manual loading and unloading of spools. Alternatives for the raw metal alkoxide precursors (particularly lanthanum, barium, yttrium isopropoxide) should be considered. These alternatives may include purchasing more fundamental forms of these materials (i.e. Y_2O_3) and transforming and purifying in-plant. Since the total capital expense for the sol-gel based scheme was much lower than the other processes ($17 million to $30+ million), there is extra capital that can be spent to try to reduce the costs by automation, etc. Finally, there may be concerns about decreasing product quality with increased throughput. It may be necessary to examine the maximum amount of material that can be put down per pass and still maintain epitaxial growth throughout the final product. The limitations of the material's crystallization rate combined with the nickel substrate's tendency to recrystallize back into random orientations will complicate the determination of the optimal settings for this process.

Subtask 6.03 Coated Conductor Development and Program Management

Discontinued effective September 30, 1999.

Subtask 6.04 Optimum Coated Conductor

Continue Subtask 6.04 as funds permit.

The remaining milestone to complete the cost/ performance study for the Sol Gel process (Subtask 6.02) will be completed under this subtask.

Subtask 6.05 Cost Performance Analysis of Potential Manufacturing Processes

Discontinued effective August 31, 1999.

Subtask 6.06 Development of Real Time process Control using In-Situ Diagnostics

Discontinued effective September 30, 1999.

OPEN ITEMS

The Draft Quarterly Technical Progress Report, for the period January 1 - March 2000

SUMMARY STATUS ASSESSMENT AND FORECAST

Environmental restoration activities at the CFFF will continue and funding to cover these activities will be continued in accordance with the SETTLEMENT AGREEMENT.

Contract reporting requirements are being met on time. Future contract reporting requirements need to be reviewed and modified in accordance with the SETTLEMENT AGREEMENT.

ANALYSIS OF VARIANCE

The variances are all negative due to the planned expenditures being zero, due to the lack of an FY-2000 Management Plan.

APRIL 1, 2000, THROUGH JUNE 29, 2000, PRELIMINARY QUARTERLY VARIANCE REPORT

Planned vs. Actual Expenditures
(thousands of dollars)

TASK	PLANNED	ACTUALS	VARIANCE
1	0.0	0.0	0.0
2	0.0	0.0	0.0
3	0.0	12.1	-12.1
4	0.0	0.0	0.0
5	0.0	0.0	0.0
6	0.0	25.9	-25.9
TOTALS	0.0	38.0	-38.0

COST ELEMENT			
DIRECT LABOR	0.0	17.7	-17.7
FRINGE BENEFITS	0.0	2.8	-2.8
EQUIPMENT	0.0	0.0	0
EXPENDABLE MATERIAL	0.0	0.0	0
OUTSIDE CONTRACTS	0.0	5.5	-5.5
TRAVEL	0.0	0.0	0
TOTAL DIRECT COSTS	0	26	-26
INDIRECT COSTS	0.0	12.0	-12
TOTAL	0.0	38	-38

Planned vs. Authorized Funding
Cumulative

Task	PLANNED	AUTHORIZED FUNDING
1	1496.4	
2	614.3	
3	558.3	
4	0.0	
5	0.0	
SUBTOTAL	2669.0	2282.7
6	3689.7	3820
TOTAL	6358.7	6102.7