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FINAL TECHNICAL REPORT

FOR

DEVELOPMENT AND TESTING OF A SUPERCONDUCTING LINK FOR AN IR DETECTOR

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I. Overview of the facilities

During this period we continued to build our laboratory measurement capability. A summary of our current facilities follows:

EXPERIMENTAL CAPABILITIES

Electrical Resistivity (20 - 300 K)
Electrical Resistivity (300 - 900 K)
ac Susceptibility (20 - 300 K)
Specific Heat (20 - 300 K)
X-ray Diffraction (20 - 300 K)
Thermal Conductivity (20 - 300 K)

EXPERIMENTAL FACILITIES

Closed-cycle refrigerator (20 - 300 K)
2 Hewlett Packard data acquisition systems with multipen
plotter for presenting results
Apple IIe data acquisition system for cycling studies
Microvoltmeters
Constant current sources
Constant voltage source
Standard resistor
Lock-in amplifiers
General Electric X-ray diffraction apparatus

PREPARATION FACILITIES

Large Marshall Tube furnace (1400 °C) for oxygen
treatment studies
Three Processing Ovens with flowing oxygen for final treatment
Muffle ovens for air treatment (1100 °C)
25 Ton Hydraulic Press for pelletizing (1/2", 1", 2" diameter dies)
Mixer/Mill for grinding powders

II. Areas of study

We have made measurements in many areas that are important to the production and characterization of a superconducting link.

A. Processing studies

We have continued to improve the quality of our sintered samples through *in situ* studies of processing of $YBa_2Cu_3O_x$. Two computer programs were written to automate the data acquisition necessary to develop the sintering process and improve the critical current. The first program measures the transport critical current density (J_c) at 77K by incrementing the source current at fixed time intervals and recording the voltage. The second program monitors the resistance of the sample during the sintering process (20 - 920° C). With these two additions to our laboratory repertoire we have been able to improve critical currents by up to 10 percent. The ability to make and characterize such improvements will be important for the final superconducting link. (See Ref. 9)

B. Electrical contacts

To have a useful superconducting link, it is necessary to make good electrical contact to the superconducting material. This is not a trivial task since the superconducting material is ceramic. We have extended our studies of a melting technique for making low-resistance contacts to high temperature superconductors. We have made contacts to both $YBa_2Cu_3O_{7-x}$ and $Bi_2BaSr_2Cu_2O_8$, and to related superconducting compounds by melting gold or silver pads onto the samples before the final oxygen treatment. Scanning electron microscope studies show that both gold and silver do not diffuse far from the contact area. The surface contact resistivity of the best contacts made by the melting technique has an upper limit value in the 10^{-8} Ω-cm^2 range at 77 K. This method of making low-resistance contacts to high superconducting transition temperature (T_c) materials can be integrated into the final oxygen treatment of many prospective superconducting elements or devices. (See Ref. 6)

C. Radiation damage

Superconducting links will have to withstand the radiation environment in space. We have investigated the effect of 1 Mev electron irradiation up to a total dose of 5.7×10^{17} el/cm^2 at room temperature on $YBa_2Cu_3O_x$ with gold bead contacts made by the melting technique. We measured the superconducting transition temperature T_c, the critical current density J_c at 77 K, the normal state resistivity, and the contact resistance for gold bead contacts as a function of fluence on the same samples without disturbing the contacts. T_c remained constant at 91 K and

J_c at 77 K remained constant around 90 A/cm^2. The normal state resistivity increased systematically by about 15% for the total dose. Finally, the surface contact resistance at 77 K remained less than 4.2 $\mu\Omega$-cm^2 throughout the radiations. These studies took place over an 8 month period and subsequent measurements indicate that the results are definitely due to radiation effects and not aging effects. Since the total dose represents 120 years of electron exposure in geosynchronous orbit, we conclude that the superconductor $YBa_2Cu_3O_x$ with gold bead contacts would perform well in a space environment of electron irradiation. (See Ref. 7)

D. Materials preparation

It is always important to investigate new materials with potential applications. The discovery of higher superconducting transition temperatures in the BiPb system has created excitement in the scientific community. To investigate this phenomenon we processed $Bi_{1.8}Pb_{.6}Sr_2Ca_2Cu_3O_x$ samples by placing them in a tube furnace at 840°C to 865°C for periods up to 17 days, removing samples periodically. We have measured the superconducting transition temperature T_c and critical current density J_c on many of the samples. We obtained superconducting transition temperatures of up to 106K with modest critical current densities of 50 A/cm^2. (See Ref. 5)

E. Cycling studies

Under actual working conditions the superconducting links will undergo temperature cycling. We set up a system to monitor the effect of such cycling on the electrical contacts, the transition temperature, and the critical current. The data acquisition system was automated using an Apple IIe computer with a MetraByte D/A-A/D interface board. Data are stored on disk after each half cycle and plotted in real time so the experimenter can monitor sample condition. Samples are mounted on a probe of electrically and thermally insulating material to prevent shorting the sample and to reduce the load on the LN_2 bath. The probe is raised and lowered by a gear driven brass rod and a stepper motor. Sample and contact resistance are measured throughout the run at LN_2 and ambient temperatures using a four-probe dc technique. The current is reversed twenty times and the signals are averaged to eliminate thermal voltages and to reduce errors from random noise. With this system signals can be resolved as small as 2 μV. The overall precision of the instrumentation is better than one percent and the accuracy of the data is correct to within approximately five percent. In a span of over six thousand cycles the silver contacts in conjunction with $YBa_2Cu_3O_x$ superconductors have shown no sign of significant deterioration. The transition temperature remained constant throughout cycling and a small, but not debilitating, degradation of the critical current was observed. Therefore the contacts and material should withstand even the most demanding applications. (See Ref. 8)

F. Aging Studies

We have measured the superconducting properties and the resistance of our contacts for six samples over a two and one-half year period. Generally, we observe no effect on T_c and only modest effects or no effect on J_c and the resistance of our contacts. The results indicate that the superconductors can hold up for considerable periods of time, which is contrary to some of the earlier reports. This is most likely due to the better quality of the material.

G. Thermal conductivity

We have developed the programs and hardware necessary to measure thermal conductivity in our closed cycle refrigerator from 20 to 120K. These measurements are crucial to the characterization of a superconducting link, which should ideally have a very low thermal conductivity. Our preliminary measurements agree with other results reported in the literature.

III. Superconducting link studies

We have studied the superconducting link from a theoretical and experimental point of view.

A. Theory

Computer programs were written to model an ideal link carrying current between two fixed temperatures (4.2 and 30K in our case). The link was assumed to be uniform. The heat load on the cryogenic fluid was calculated and compared for two configurations: a harness of #40 manganin wires planned for the SAFIRE project and 1 mil by 4 mil BAYCO superconducting film strips deposited on a 1 mil thick Yttrium stabilized ZrO_2 substrate. The superconducting link yields a heat load reduction of about a factor of two. This could result in an increased mission lifetime of approximately a factor of two in cases where the link is a major contributor to the heat load, such as the envisioned two-stage cooling systems for future missions.

B. Experiment

We have worked to improve the critical currents in the tape cast materials provided by Clemson University. In their BAYCO superconducting samples containing silver, we have been able to obtain improvements of a factor of 7 in the critical current through further processing. We have also obtained thick film samples from Electro Science Laboratories. These samples show promise because they are of approximately the right dimension for a superconducting link that could be used in a project like SAFIRE, multiple strips can easily be layed down, and the films can be deposited on Yttrium stabilized ZrO_2. The samples we have received to date have a wide transition temperature of $\approx$ 50K with an onset at $\approx$ 90K. The company has produced samples with high critical currents ($\approx$200 A/cm^2) at 77K in the past and it is just a matter of time until they fine tune their process to produce such samples with our geometrical requirements.

IV. Summary of Achievements

A. Better quality high T_c material through improved processing.

B. High quality electrical contacts (10^{-8} Ω-cm^2) to high T_c material.

C. Studies on effects of electron irradiation, temperature cycling, and aging on superconducting properties indicate materials will be suitable for space applications.

D. Partnership with Electro Science Laboratories to fabricate and characterize a superconducting link by measuring T_c, J_c, and thermal conductivity.

APPENDIX A: SUPERCONDUCTIVITY RESEARCH AT CHRISTOPHER NEWPORT COLLEGE

Date	Research
March 1987	Seminal meeting of APS - open discussion of high Tc materials
May 1987	Prepared first $YBa_2Cu_3O_x$ with T_c = 92K and exhibiting a Meissner effect
July 1987	Established superconductivity lab at CNC
Fall 1987	Developed noble metal, low-resistance contacts to $YBa_2Cu_3O_x$
Fall 1987	Prepared $YBa_2Cu_3O_x$ at 90% theoretical density
January 1988	Attained critical current density of ≈200 A/cm^2 in $YBa_2Cu_3O_x$
1988-89	Electron radiation damage studies on $YBa_2Cu_3O_x$ and contacts
Summer 1989	Measurement of thermal conductivity in $YBa_2Cu_3O_x$
1988-90	*In situ* resistivity studies while processing high T_c superconductors
Spring 1989	Prepared BiCaSrCuOPb alloys with T_c = 106K
1989-1990	Characterized Clemson $YBa_2Cu_3O_x$ samples for possible use as electrically conducting, thermally isolating link in SAFIRE type project
Spring 1990	Room temperature to 77K cycling studies on $YBa_2Cu_3O_x$ and contacts

APPENDIX B: Presentations

1. "The Effects of Repeated Cycling from Liquid Nitrogen to Ambient Temperature on High Tc Superconductors"; J. Beaufait, R. Caton, and R. Selim; presented at the Fourth National Conference on Undergraduate Research, April 19-21, 1990, Union College, Schenectady, NY.

2. "Improved Sintering Process of Superconducting $YBa_2Cu_3O_x$"; R. W. McKitrick, Jr., R. Selim, and R. Caton; presented at the Fourth National Conference on Undergraduate Research, April 19-21, 1990, Union College, Schenectady, NY.

3. "The Effects of Repeated Cycling from Liquid Nitrogen to Ambient Temperature on High Tc Superconductors"; J. Beaufait, R. Caton, and R. Selim; presented at the Sixty-Eighth Annual Meeting of the Virginia Academy of Science, May 23-26, 1990, George Mason University, Fairfax, VA.

4. "Improved Sintering Process of Superconducting $YBa_2Cu_3O_x$"; R. W. McKitrick, Jr., R. Selim, and R. Caton; presented at the Sixty-Eighth Annual Meeting of the Virginia Academy of Science, May 23-26, 1990, George Mason University, Fairfax, VA.

APPENDIX C: List of Publications

1. "Rugged Low-Resistance Contacts to $YBa_2Cu_3O_x$"; R. Caton, R. Selim, A. M. Buoncristiani, and C. E. Byvik. Appl. Phys. Lett. **52**, 1014 (1988).

2. "Rare Earth Substitution Studies of the High Temperature Superconductor $YBa_2Cu_3O_x$"; W. Edwards, R. Harvey, A. Plum, S. Yang, R. Selim, and R. Caton; Proceedings of the Second National Conference on Undergraduate Research, University of North Carolina at Ashville, J. M. Sulock and D. L. Seymour, Eds., P. 395, (1988).

3. "High Temperature Resistivity of the $YBa_2Cu_3O_x$ Superconductor"; B. Almeida, F. Tambone, R. Caton, and R. Selim; Proceedings of the Second National Conference on Undergraduate Research, University of North Carolina at Ashville, J. M. Sulock and D. L. Seymour, Eds., P. 347, (1988).

4. "Frequency-Dependent Ultrasonic Attenuation of $YBa_2Cu_3O_7$"; K. J. Sun,W. P. Winfree, M. K. Xu, Bimal K. Sarma, M. Levy, R. Caton, and R. Selim. Phys. Rev. B **38**, 11988 (1988).

5. "In Situ Resistivity Measurements of the BiPb System During Processing as a Predictor of Superconducting Properties"; B. Almeida, K. Anthony, R. Caton, and R. Selim. Proceedings of the Third National Conference on Undergraduate Research, Vol. 1 K. M. Whatley , Ed., Trinity University, San Antonio, P. 161 (1989).

6. "Low-Resistance Noble Metal Contacts to High-Temperature Superconductors"; R. Selim, R. Caton, A. M. Buoncristiani, C. E. Byvik, R. A. Edahl, Jr. , and S. Wise. J. Appl. Phys. **67**, 376 (1990).

7. "The Effect of Electron Irradiation on $YBa_2Cu_3O_x$ with Gold Bead Contacts"; R. Caton, R. Selim, A. M. Buoncristiani, and C. E. Byvik; J. Appl. Phys. **67**, 7478 (1990).

8. "The Effects of Repeated Cycling from Liquid Nitrogen to Ambient Temperature on High Tc Superconductors"; J. Beaufait, R. Caton, and R. Selim; Proceedings of the Fourth National Conference on Undergraduate Research, Vol. 1, K. M. Whatley , Ed., Union College, Schenectady, New York, p. 241 (1990).

9. "Improved Sintering Process of Superconducting $YBa_2Cu_3O_x$"; R. W. McKitrick, Jr., R. Selim, and R. Caton; Proceedings of the Fourth National Conference on Undergraduate Research, Vol. 1, K. M. Whatley , Ed., Union College, Schenectady, New York, p. 247 (1990).

10. "Rugged Low-Resistance Contacts to High Tc Superconductors", R. Caton, R. Selim, C. E. Byvik, and A. M. Buoncristiani, NASA Tech Briefs, LAR-13964, accepted for publication.