NASA Technical Memorandum

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CHARACTERIZATIONS OF ELECTRICAL PROPERTIES OF HIGH T_c SUPERCONDUCTING MATERIALS

Center Director's Discretionary Fund Final Report

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Science and Engineering Directorate

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I. INTRODUCTION

The goal of this project is to develop a data acquisition system that will automatically characterize transition temperatures (T_c) and their widths, critical current densities (J_c), and magnetic properties of thin films and bulk samples of high T_c materials as a function of temperature. While hardware already exists for making current-voltage (I-V) traces of niobium thin film microbridges, much higher currents are carried by bulk high T_c samples even when sliced into small cross sections. In addition to requiring larger currents for driving the superconducting sample normal, the existing hardware is not easily adaptable for computer interfacing.

The new hardware consists of three current sources, each selected to cover a specific range of current at a particular resolution. The system also uses a nanovoltmeter to measure the voltage across the sample and a multiplexed, ten-channel multimeter for measuring either the sample voltage or the platinum temperature sensor. During superconducting transition, a multi-frequency inductance meter measures the change in inductance of a small test coil wound around the high T_c sample. Each instrument's sequence of events is smoothly controlled by a computer, eliminating human error and fatigue involved in making a large number of repetitious measurements.

II. VOLTAGE MEASUREMENT APPROACH

The high T_c sample resistance is calculated from the carefully measured voltage across a length of sample divided by the current passing through the sample. Typical voltages measured across a bulk high T_c sample may vary from zero volts (superconducting state) to a few millivolts (normal state) depending on the current passing through the sample. High T_c critical currents through a 2 mm^2 cross sectional area of sample may vary from a few milliamperes to tens of amperes depending on sample quality and measurement temperature.

To measure vanishing small voltages down to background levels of the system (±35 nV), a four-wire measurement approach must be used in order to cancel thermally induced voltages and eliminate lead resistance. With the sample cooled to 77 K, a thermally induced error voltage of 26 μV is produced on this system offsetting any small voltages completely off scale. Thermal voltage error can be canceled by measuring not one voltage but two voltages, provided they are made before the thermal gradients change. The first voltage (V_1) is measured across the sample while the current (I) flows in one direction. The second voltage (V_2) is measured across the sample while the same magnitude of current flows in the opposite direction. The corrected voltage (V_c) and corrected resistance(R_c) are given by:

$$V_c = (V_1 - V_2)/2, \qquad R_c = V_c/I.$$

III. TEST FIXTURE

High T_c materials typically have rough surfaces that are difficult to wet with solder. These poor contact areas may result in unacceptable levels of ohmic heating at higher current values. One satisfactory sample connection and mounting technique involves the ultrasonic soldering of indium (99.99 percent pure) pads to the upper and lower surface of each end of the sample. The high T_c sample is then mounted into a indium-filled copper test fixture raised to a temperature slightly above the melting point of indium. The sample's ultrasonically bonded indium melts on contact with the hot test fixture, forming good contacts between the sample and current contacts. The test fixture consists of a double-sided circuit board sandwiched between two copper blocks soldered together with higher melting point solder. The higher temperature solder remains solid and keeps the two copper blocks bonded together while the sample can be mounted in or removed from the molten indium. Each copper block has a deep channel milled for holding a small reservoir of indium and the sample. Current leads are attached to each side of the test fixture by bolt-on lugs, and indium voltage lead pads are ultrasonically attached to the sample about 1 mm apart. Magnetic measurements are made by winding a small test coil around each sample located between the two voltage pads. A photograph of the test fixture complete with the high T_c sample and four-point probe electrical connections is shown in Figure 1.

IV. DATA ACQUISITION

Instrument control, data acquisition, and data reduction by computer are essential for an acquisition system to operate with minimum human interaction. The computer chosen for this project is the Hewlett Packard Model 310 Low Cost Color Workstation. Built into the workstation is a HP-IB (IEEE-488) interface for communicating with the voltmeter, current sources, inductance meter, multimeter, printer, plotter, and disc drive. The system voltmeter is a Keithley instruments model 181 nanovoltmeter. Two of the three current sources used in the system are also Keithley instruments, models 220, and 228. The multimeter, inductance meter, and other current source are Hewlett Packard instruments models 3457A, 4275A, and 6031A. A photograph and schematic of the complete data acquisition system is shown in Figures 2 and 3, respectively.

The data acquisition software was written in-house and allows three types of measurements to be performed. The first type of measurement, the current-voltage (I-V) trace, varies the current passing through the superconducting sample in discrete known values. At each value of current the temperature is measured and the voltage across the sample is measured using the technique described earlier. The information obtained at each point is displayed graphically on the screen in real time. The voltage will remain essentially zero, except for background noise, as long as the sample is superconducting. As soon as the current exceeds a critical value, known as the critical current (I_c), voltage across the sample begins to increase with increasing current. Some time later the (I-V) trace is stopped and all current, voltage, and temperature data are stored on disc for plotting at a later time. A typical (I-V) trace is shown in Figure 4. The sample is submerged in a bath of either liquid helium (4.2 K) or liquid nitrogen (77 K) during this measurement.

The second type of measurement, the resistance-temperature (R-T) trace, passes a small constant excitation current through the high T_c sample while the temperature is varied. A range of temperatures is obtained by first dipping the sample into a liquid nitrogen bath and then raising the sample slightly above the liquid level after the sample has completely cooled. As the sample slowly warms, voltage, magnetic, and temperature measurements are made every few seconds and displayed in real time. The critical temperature (T_c) is determined by these data. After the trace is complete the information is stored for later use. A typical (R-T) trace is shown in Figure 5.

The third type of measurement records the magnetic behavior of the sample during the resistance-temperature trace. A small coil of copper wire, about 15 turns, wound around each sample gives about 1.5 μH inductance at room temperature. As the sample cools through the transition temperature, magnetic field is expelled and shielded by the superconductor causing a change in measured inductance of the coil. The quality of a high T_c superconductor can be inferred by looking at the transition width of the trace. The sharper the transition the better the sample. In multiphase superconducting compounds, this type of measurement can show the transition temperature associated with each phase. A typical trace of the magnetic measurement is shown in Figure 6.

V. RESULTS

The high temperature superconductivity data acquisition system performs extremely well. Noise for the system is very low, fluctuating on average only ±35 nV for a superconducting sample at 77 K. Over 250 measurement traces have been performed on 70 different high T_c samples thus far. Current-voltage data were obtained and published in a paper on the enhanced properties of silver oxide doped $YBa_2Cu_3O_x$ compounds [1].

VI. CONCLUSIONS

Even though the system performs well, there are two areas under improvement. Presently resistance-temperature measurements are limited from room temperature down to 65 K. Below this value the temperature sensors are not calibrated. Currently the temperature rate of change and range during a resistance-temperature measurement is controlled by raising the sample out of a liquid nitrogen bath. The greater the sample height above the liquid level the faster the warmup. This scheme provides no control for holding long periods of time at a fixed temperature. Both of these shortcomings will be alleviated with the addition of a closed cycle helium refrigerator system. This refrigerator system can control the temperature from 300 K to 10 K and incorporates two calibrated silicon diodes for measuring temperature over this complete range.

VII. REFERENCE

1. P. N. Peters, R. C. Sisk, E. W. Urban, C. Y. Huang, and M. K. Wu, Appl. Phys. Lett. , 52 , 2066 (1988).

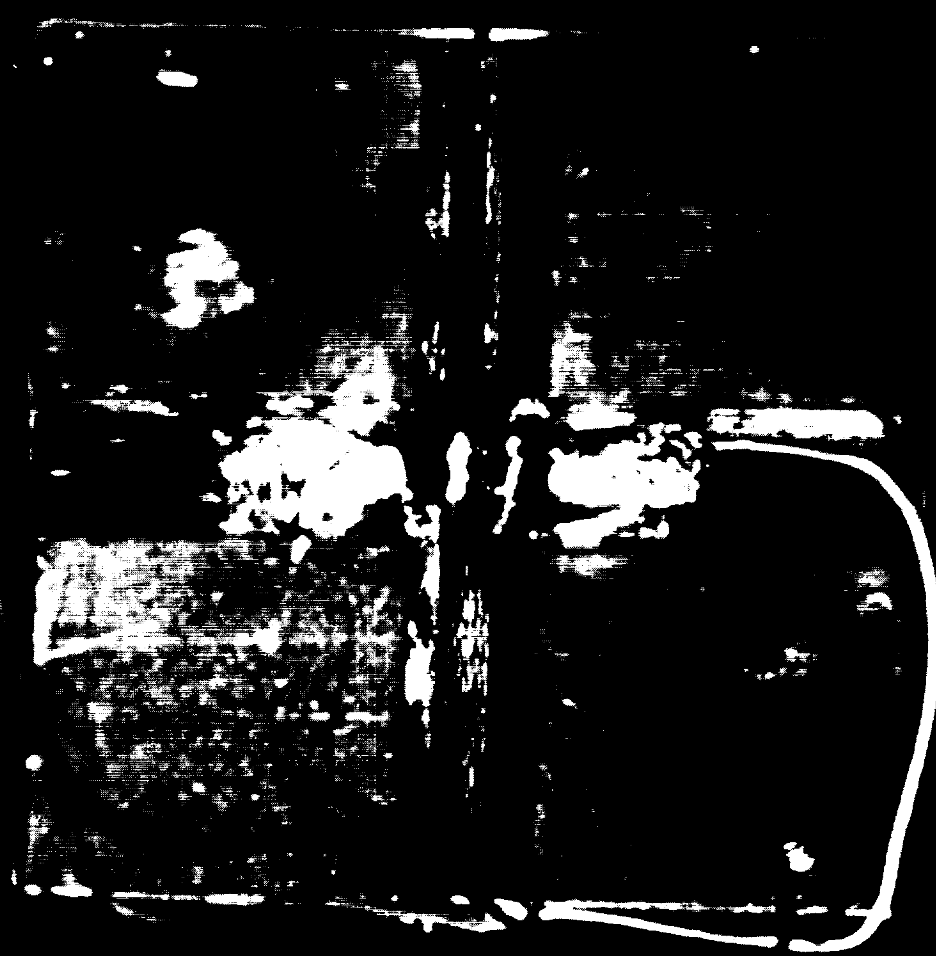

Figure 1. Test fixture.

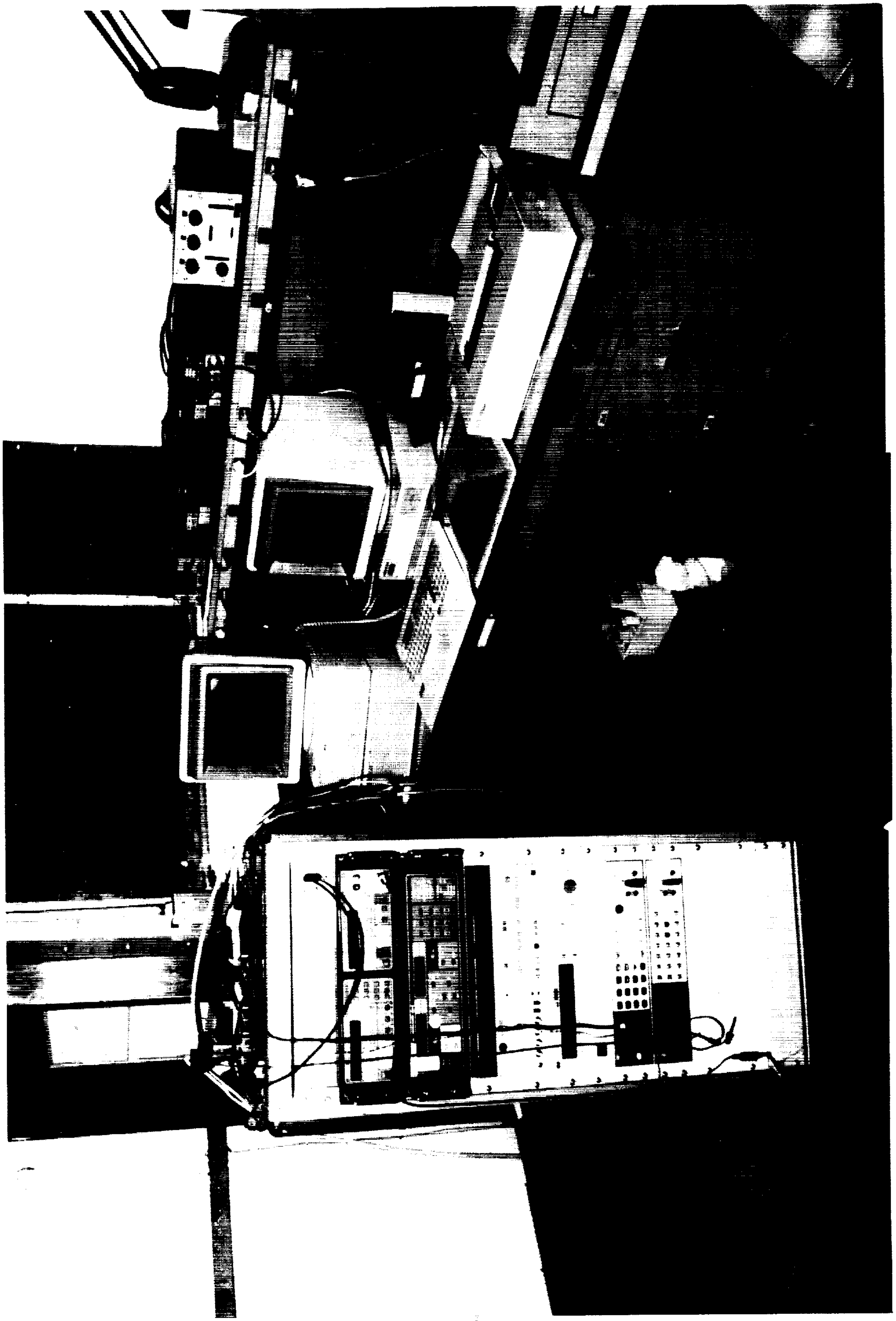

Figure 2. Data acquisition system.

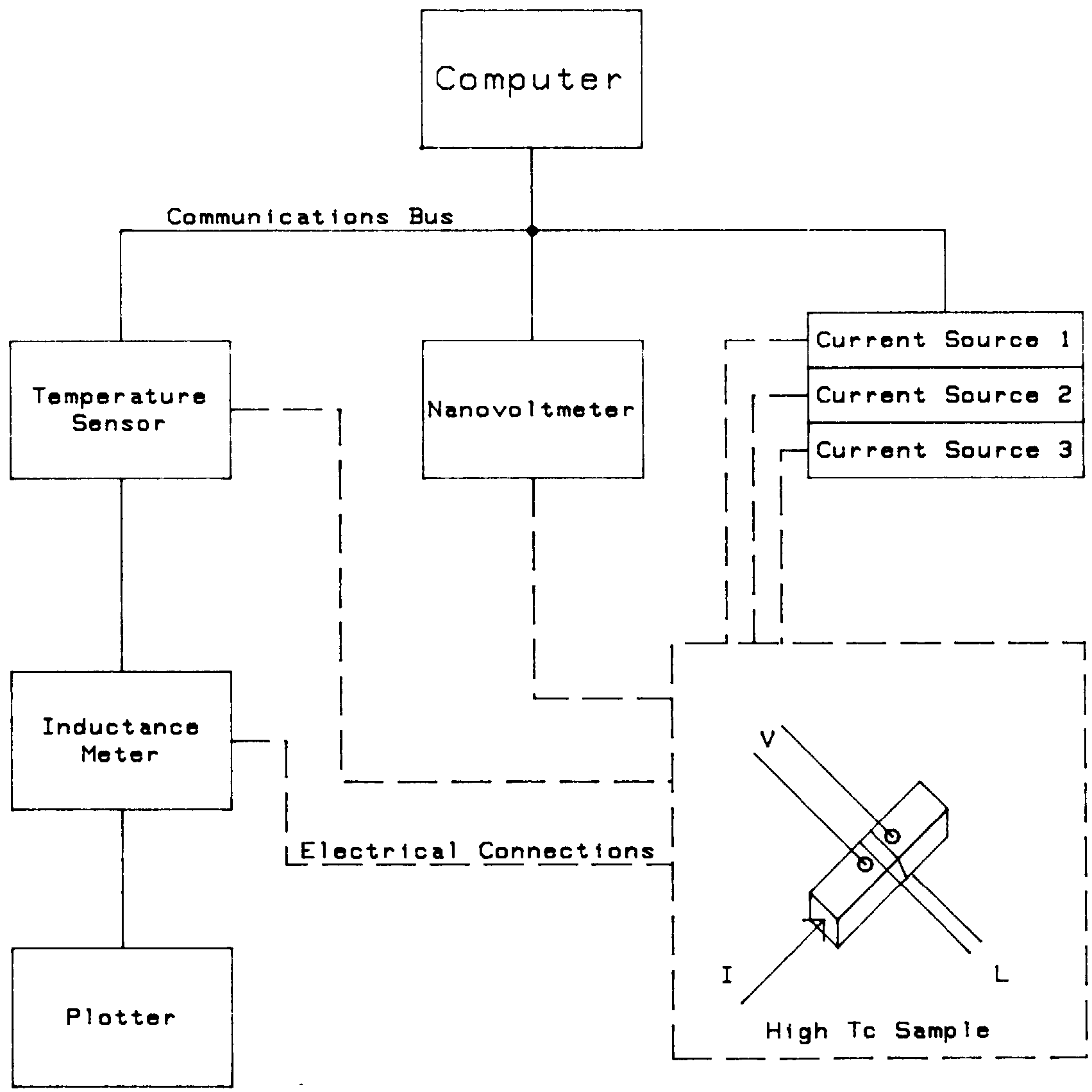

Figure 3. Schematic of the data acquisition system

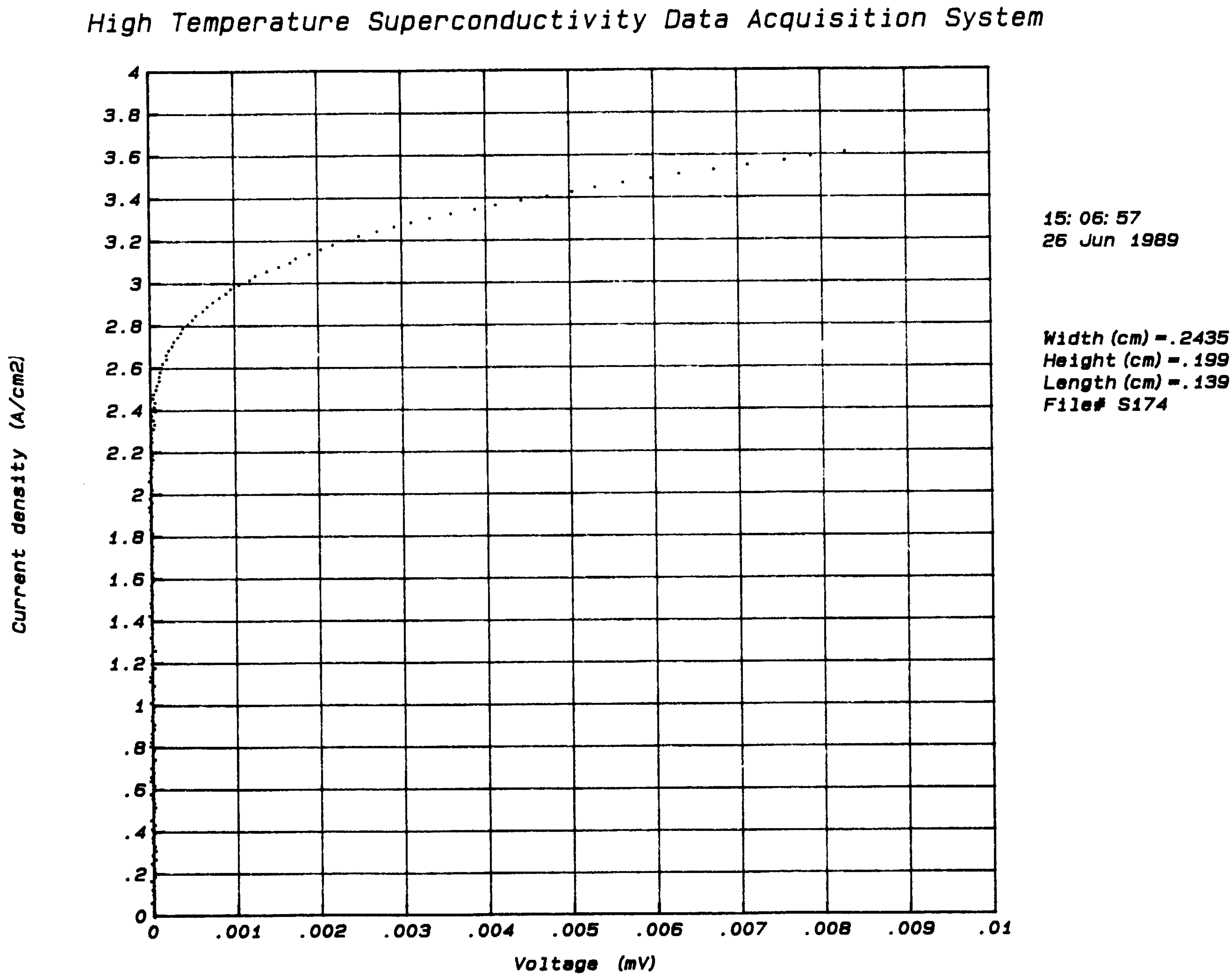

Figure 4. Typical current-voltage trace.

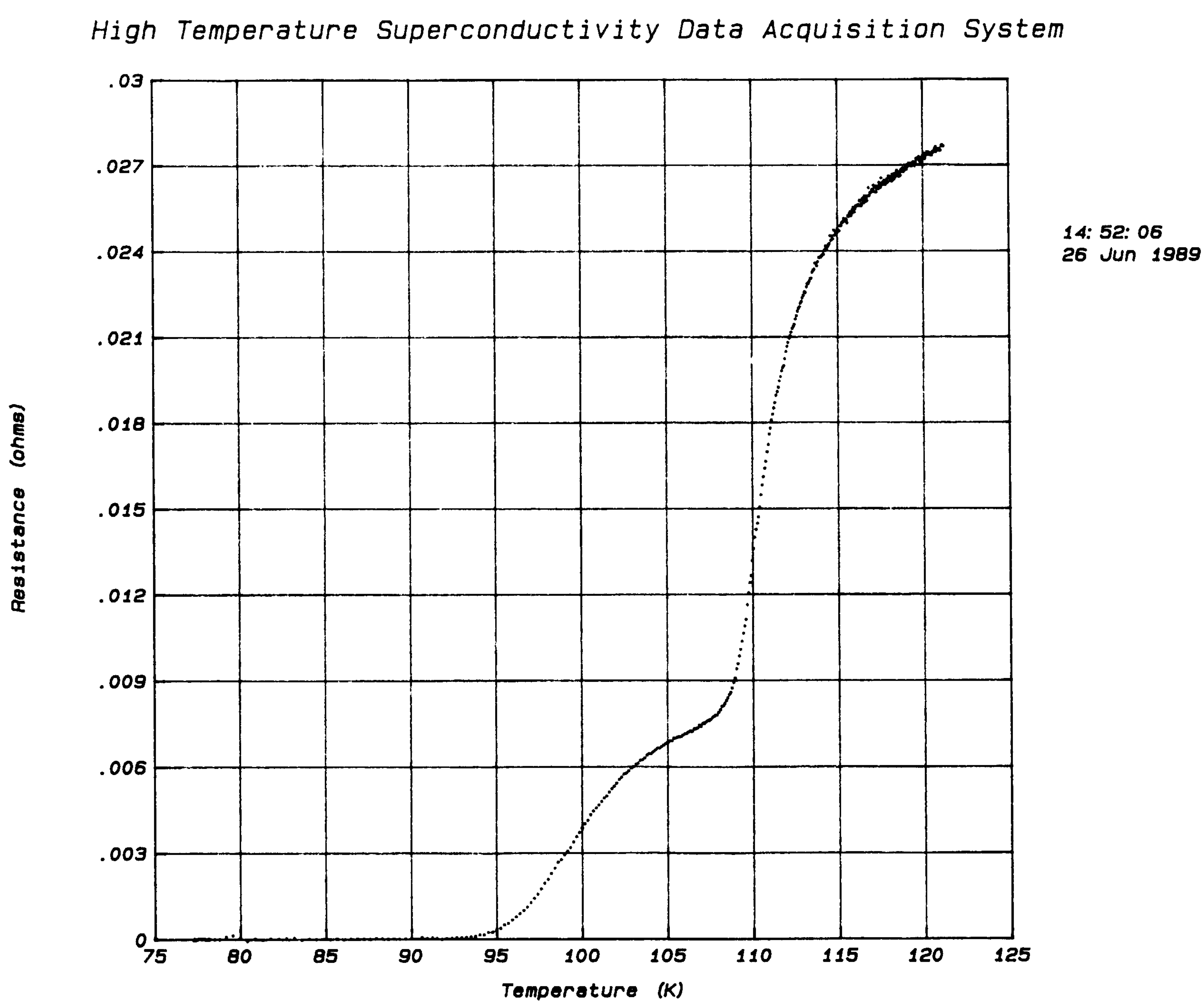

Figure 5. Typical resistance-temperature trace.

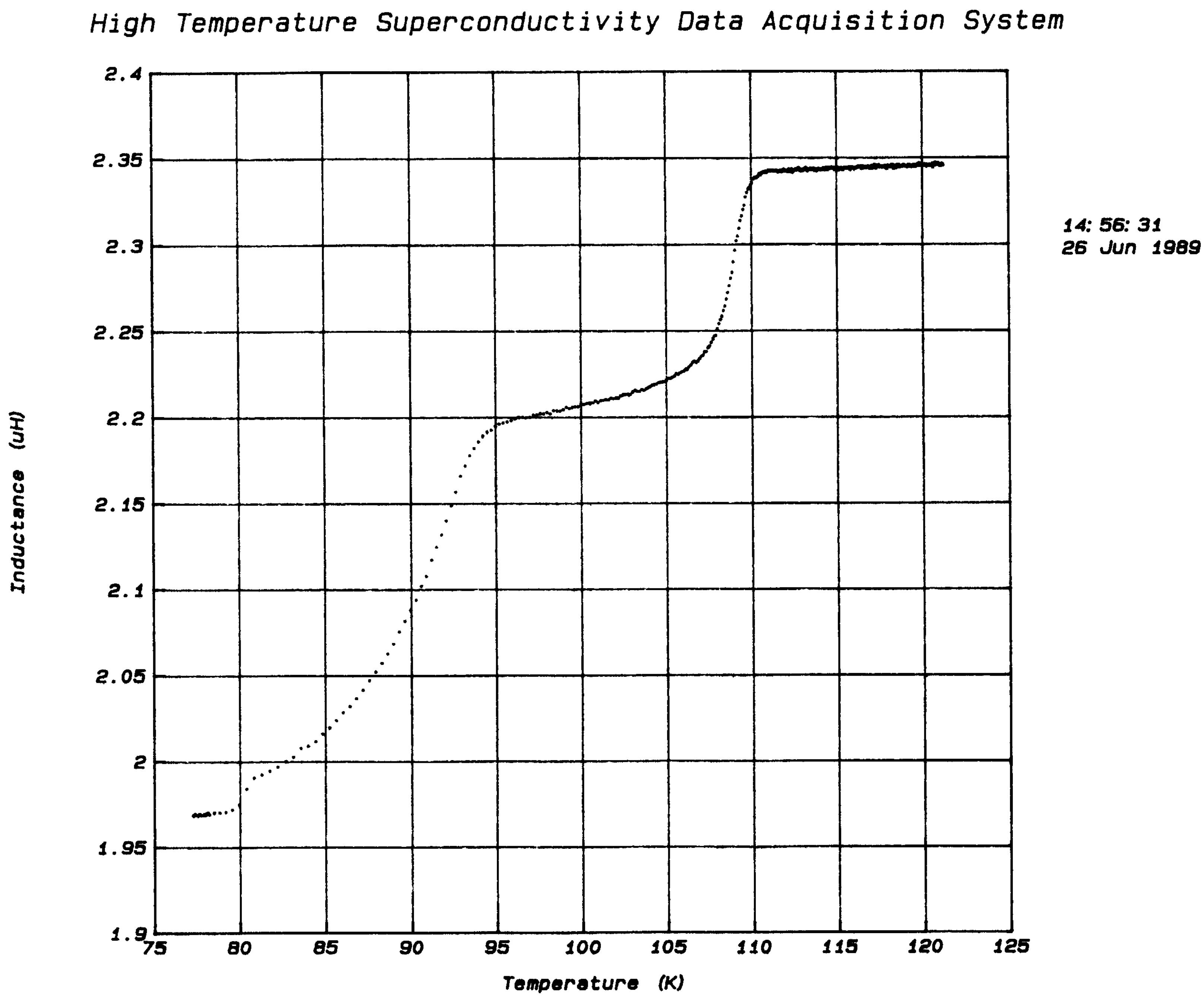

Figure 6. Typical magnetic measurement trace.

APPROVAL

CHARACTERIZATIONS OF ELECTRICAL PROPERTIES OF HIGH T_c SUPERCONDUCTING MATERIALS— CENTER DIRECTOR'S DISCRETIONARY FUND FINAL REPORT

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The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

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Director
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16. ABSTRACT

This report describes the automated data acquisition system developed in the Space Science Laboratory at Marshall Space Flight Center for measuring electrical properties of high temperature superconductors. The acquisition system, consisting of a computer and computer-controlled hardware, allows large numbers of voltage, current, temperature, and magnetic measurements to be performed on bulk and thin film samples. Typical results are shown characterizing transition temperature (T_c), critical current density (J_c), and magnetic properties of bulk high T_c materials as a function of temperature.

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