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Application of a Microcomputer in Resistivity Measuring System

(microcomputer/instrument/electrical conductivity)

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An automatic digital system for measuring the temperature dependence of the electrical resistivity is described. A 16-bit microcomputer is employed for controlling experimental apparatus, reading data reading data from a digital voltmeter, processing, storing data on a floppy disk, and presenting the computed results in forms of graph. Three-and two-channel computer-controlled switches can be construced by utilizing an output interface board and relays.

INTRODUCTION

In a last few years there has been growing interest in synthesizing quasi two- or one-dimensional materials. Those materials exhibit interesting physical phenomena which cannot be observed in the three-dimensional ones.[1,2] Especially, some of the low-dimensional compounds undergo a structural phase-transition accompanying the formation of charge-density-waves or spin-density-waves at low temperatures. In order to know whether a newly grown compound shows such a transition or, more generally, to reveal its physical nature, electrical measurements must first be done.

The structural phase-transition usually induces an anomaly in the temperature dependence of the electrical resistivity.[2] The appearance of the anomaly can be more easily detected by investigating the temperature derivative of the resistivity as a function of temperature. In practice, the temperature derivative is not obtained by direct measurement but by computation, which needs a lot of data collected with a small temperature interval. In such a case, an analog data system is clearly inconvenient, and a digital data system is useful because data can be handled in calculation with the help of a computer. In addition, the latter system with automatic operations eliminates the repetition of the inefficient manual operation which is intolerable in most cases.

We have developed a digital data collection and control system which is operated by a microcomputer. The automatic instrumentation system involves control of experimental apparatus, data acquisition, data processing and presentation of the final results in seveal forms of graph.

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AUTOMATIC INSTRUMENTATION SYSTEM

Our method for measuring the electrical resistivity is based on the four-probe method. A constant current is fed to the two contacts on a specimen by a dc constant-current supply (Yokogawa: type 2555) and the induced voltage drop across the other two contacts is measured by a digital volt-meter (DMM) (Keithley: model 195). The DMM can be operated in the autoranging mode and has the resolution of 0.1 μV in the range of 20 mV. The specimen is mounted in a cryostat and can be heeted or cooled between 20 and 330 K by use of a heater or a closed refrigeration sytem (Osaka Oxygen Industries Co.: Cryo-Mini D). A Au (+0.07 at % Fe)-Chromel thermocouple is used to determine the temperature of the specimen.

The block diagram of our automated system is shown in fig. 1. The main component in the system is a 16-bit microcomputer based on *i*8086 CPU (Central Processing Unit).[3] The microcomputer contains many peripheral devices: RAM (Random Access Memory) and ROM (Read Only Memory) with a BASIC interpreter (Beginner's All Purpose Symbolic Instruction Code), parallel input/output ports for communication with two floppy disks, external apparatus, a printer, an XY-plotter and IEEE-488 i. e. GP-IB (General Purpose Interface Bus) device, and serial input/output ports for communication with a CRT (Cathode-Ray Tube) display. The IEEE-488 bus is an instrumentation data bus with standards adopted by IEEE (Institute of Electrical and Electronic Engineers). The signal lines of the IEEE-488 bus are grouped into three categories; datalines, handshake lines and bus management lines. Through the bus, the computer gives commands to the DMM or receives data sent by it. We have attached an interface board (Nippon Electric Co.: PC-9801-19) to the computer as IEEE-488 bus interface.

Three kinds of voltages to be measured come from the specimen, the standard resistor and the thermocouple. Therefore, a 3-channel multiplexing switch is used as an input selector. A 2-channel switch is also used to reverse the direction of the current fed to the specimen. These switches have been constructed as a 5-channel switch, which can be randomly controlled by the computer, by utilizing an output interface module(Contec Co.: type PO-32)[4] and relays having mercury-wetted contacts. The relays are energized by dc 12 V.

Figure 2 shows the program flow for a resistivity measurement run. The parameters include the specimen geometry and the temperatures at which the sequence of the data acquisition is allowd. Since, normally, the specimen is cooled or warmed very slowly, we fix the first and the last temperature (T_1 and T_2), and the temperature interval (ΔT_0) for measurements. The computer first connects the voltage from the thermocouple to the DMM to determine the temperature of the specimen. If the temperature is lowered or raised by ΔT_0 than T_1 or the preceding one, the computer allows the sequence of data acquisition involving the temperature of the specimen, the voltage drop V_+ and V_- (the direction of the current is reversed) across the specimen, and the voltage of the standard resistor. These data are combined to calculate the pair of the resistivity and the temperature, which is stored on a floppy disk and is plotted in a graph on the CRT. This procedure is repeated. After measurement is completed at the temperature T_2, all the data are transfered from the floppy disk to CPU and the temperature derivative of the resistivity is computed by the following method. First, an optimum functional form of the resistivity is determined as a function of temperature from eight pairs of data by the least squares method. Then, the function is differentiated with

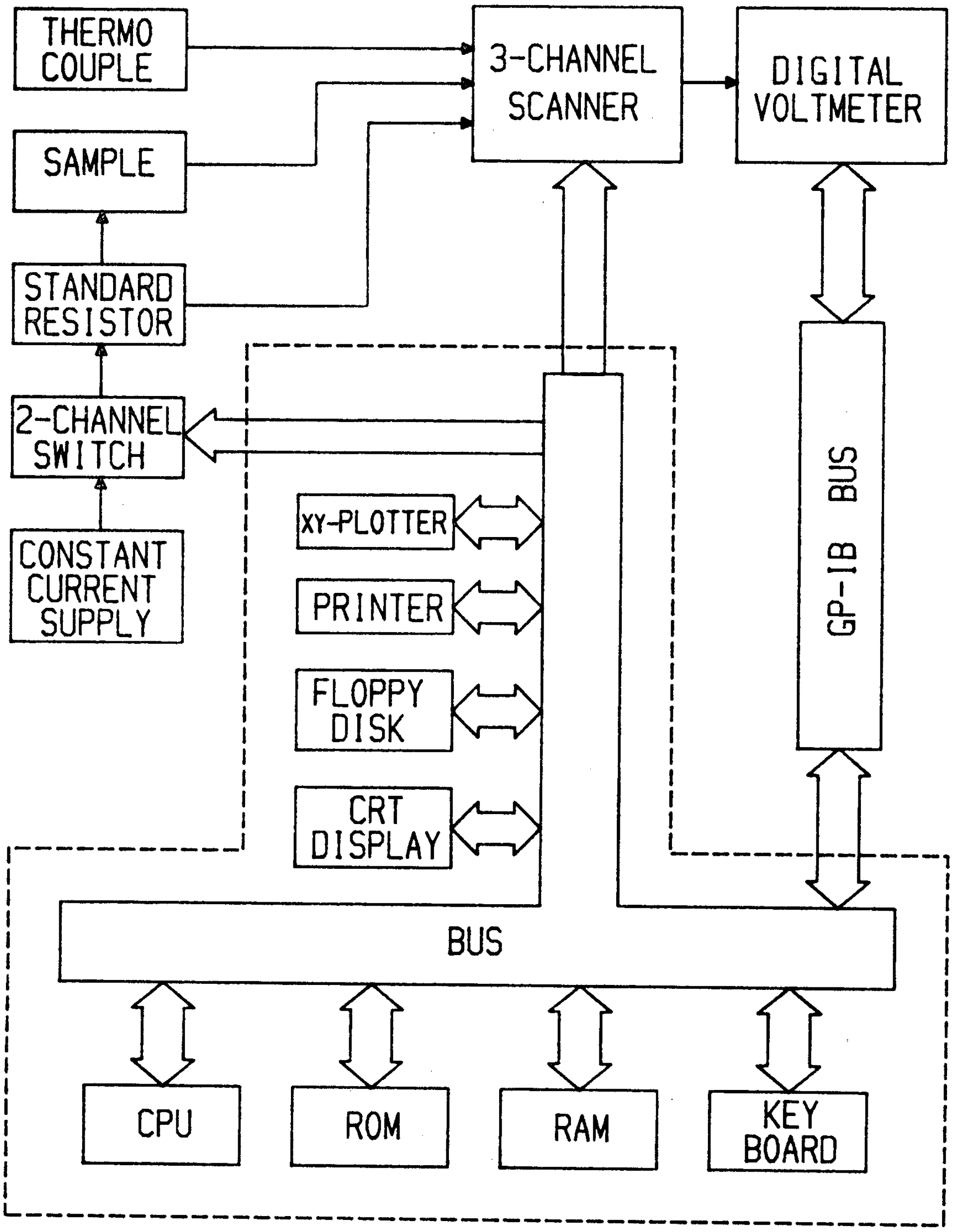

Fig. 1. Block diagram of the automated resistivity-measuring system.

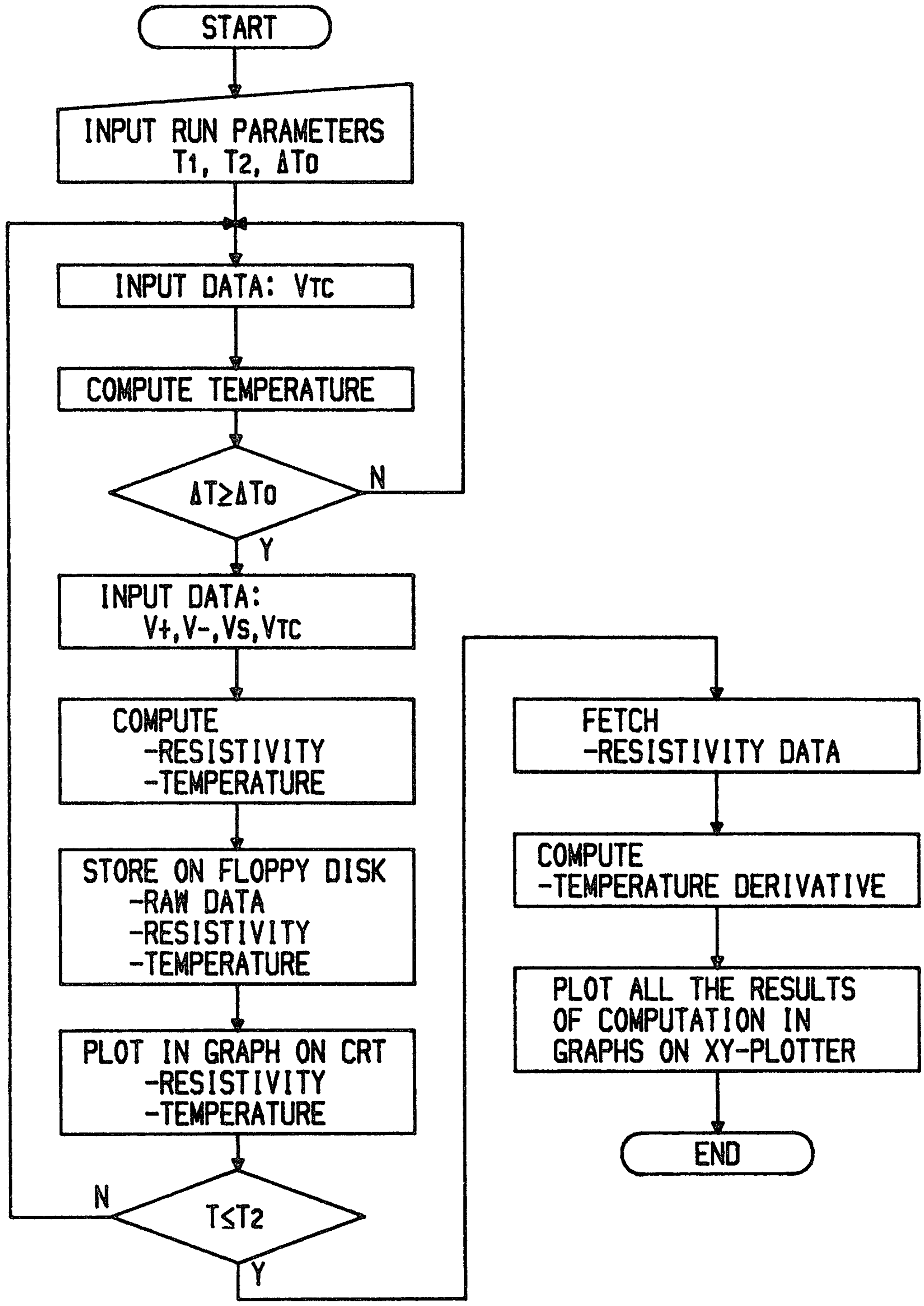

Fig. 2. Program flow graph for a resistivity measurement run. In the figure, V_+ and V_- represent the sample voltages, and V_{TC} and V_S indicate the voltage of the thermocouple and of the standard resistor.

respect to temperature. Finally, the obtained results are displayed on the CRT and printed in various kinds of graph on the X-Y plotter ; R vs T, dR/dT vs T, $\log R$ vs $1/T$ and $d(\log R)/d(1/T)$ vs $1/T$.

RESULTS OF RESISTIVITY MEASUREMENTS

By use of the digital recording-system described in the preceding section, we have measured the temperature dependence of the electrical resistivity of

$(NbSe_4)_3I$ and $(TaSe_4)_2I$. These compounds are new materials recently synthesized and are considered to be quasi one-dimensional materials.[5] Figure 3 shows the temperature dependence of the resistance of $(NbSe_4)_3I$ in the actually plotted form which includes about nine hundred data-points. Figure 4 represents the temperature derivative of the resistance. The experimental data have been collected with the cooling speed of about 0.8 K/min in average under the condition of the constant current of 10 μA and every temperature interval of 0.3 K. As temperature is decreased from 330 K, the curve becomes nearly flat at about 298 K, and starts to re-increase below 230 K. The log plot of the resistivity against the reciprocal temperature reveals two types of semiconducting behaviour above 298 K and below 230 K. The sudden decrease of the activation energy seems to be caused by the change of the space group of the compound.[6] In fig. 4, we have found a discontinuous change at 273 K in the temperature derivative curve of the resistivity.[7] The origin of the new anomaly has not been explained yet. This example demonstrates that the presence of any resistivity anomaly is easily discovered by investigating the temperature derivative of the resistivity and that our system

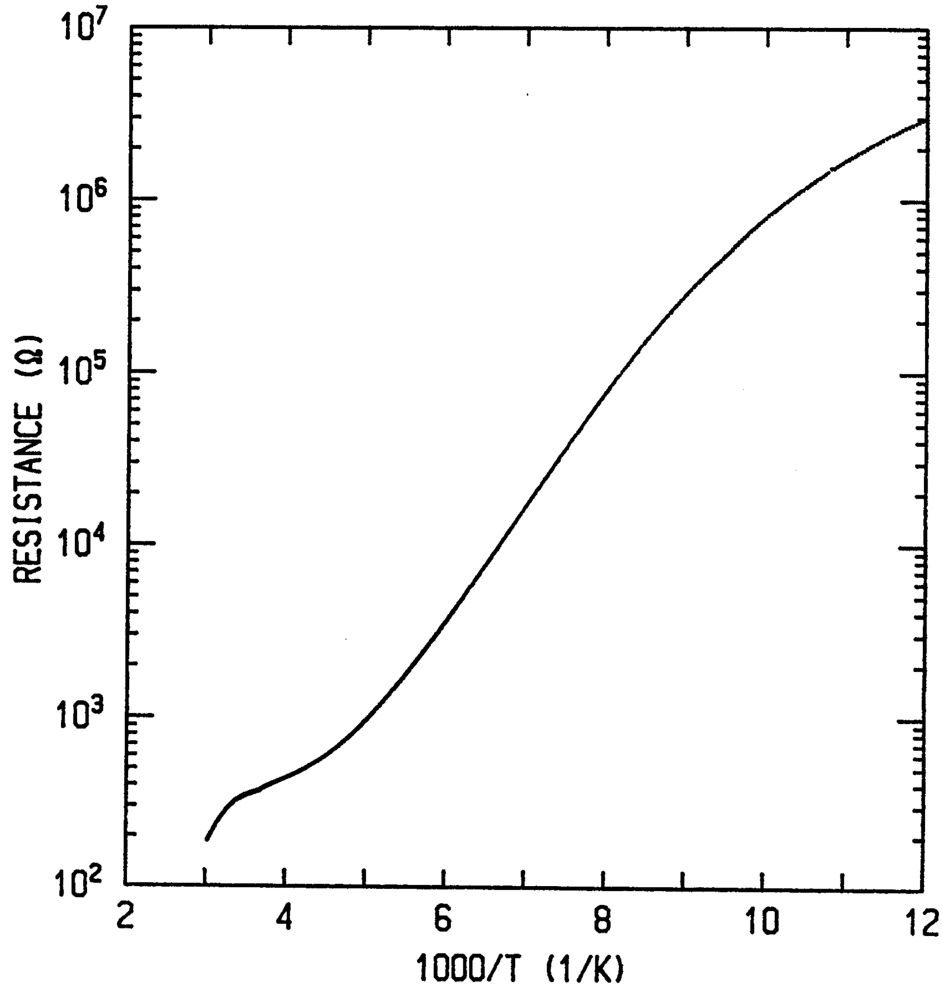

Fig. 3. Temperature dependence of the resistance of $(NbSe_4)_3I$.

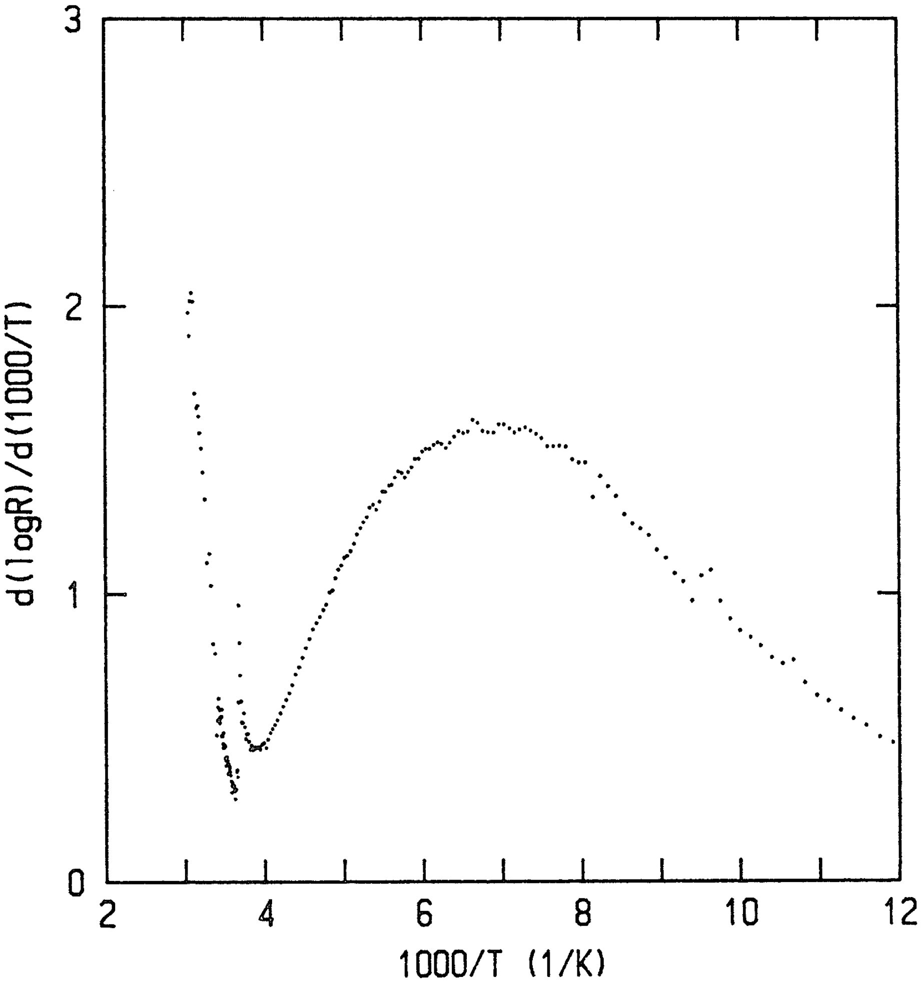

Fig. 4. Temperature derivative of the resistance of $(NbSe_4)_3I$ as a function of temperature.

is helpful in studies of electrical properties of the low-dimensional materials.

CONCLUSION

We have applied a 16-bit microcomputer to an instrumentation system for electrical measurements. The computer plays important roles in control of experimental apparatus, data acquisition, processing and presentation of the computed results in graphs. The system can be full-automatically operated with the help of the refrigerating machine in the temperature range of 20—340 K. The described digital system has been successfully used in studies of the temperature dependence of the resistivity for quasi one-dimensional compounds. The 5-channel multiplexer in the measuring system has been constructed by combining the output interface board and relays.

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