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PC Based Data Acquisition System for Resistivity and TEP Studies at High Pressures
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Abstract

In this paper we describe the development of a PC based data acquisition system for high resolution measurement of resistivity and absolute thermopower (TEP) of conducting solids at high pressures. The main feature of this system is the software PID control of temperature with stabilities of the order of ± 0.02°C in the truly hydrostatic teflon thermopower cell (temperature range 0-300°C) and ± 0.1°C in the quasi-hydrostatic high pressure - high temperature cell (temperature range 0-1000°C). A significant increase in the signal to noise ratio has been achieved by adopting real time digital filtering of the data. Linear rate of heating of the sample which is software selectable makes this system useful for other studies such as high pressure DTA.

1 Introduction

Thermoelectric power (TEP) or the Seebeck coefficient, S, is the most sensitive among the electronic transport properties of a conducting material. It is well known that experimental measurement of the temperature dependence of the TEP provides valuable data on the energy dependence of the density of states and the carrier relaxation time at the Fermi energy.¹ In the literature several analog electronic circuits have been described for the measurement of TEP as a function of mean temperature.² ⁴ In high pressure studies using the piston-cylinder apparatus, the teflon cell technique⁵ has been extensively used for generating pressure upto 50 kbar. Using this basic arrangement, several techniques for the measurement of physical properties like electrical resistivity,⁶ ⁷ thermopower,⁷ Hall coefficient,⁸ and Curie temperature⁹ ¹⁰ have been developed. The main advantage of the teflon cell technique is that the use of liquid (petroleum ether, silicone fluid, n-pentane, isomyl alcohol etc..) as the pressure
transmitting medium ensures a near hydrostatic environment for the sample under study. The use of an internal heating arrangement facilitates the measurement of the property upto 300°C. For high temperature studies upto 1000°C under quasi-hydrostatic pressure conditions the cell comprising of talc, pyrophyllite and graphite assembly can be utilised for TEP and AC resistivity studies. The development of a precision temperature controller/programmer for the teflon cell by the authors has facilitated the measurement of several physical properties under controlled conditions of temperature and pressure.

In this paper we discuss the design and performance of a PC based data acquisition system (DAS) for high resolution TEP and AC resistivity measurements at high pressures and temperatures. Software control of temperature through a PID algorithm with provision for fine tuning of the parameters and real time digital filtering of the data (improved signal to noise ratio) have led to a significant improvement in the resolution of the TEP and AC resistivity measurements. The performance of the system is demonstrated by presenting some typical data on pressure induced continuous phase transitions.

2 Teflon cell arrangement

In this section we briefly describe the teflon cell technique with special reference to the arrangement for thermoelectric studies. However the cell arrangement can be easily modified for resistivity and Hall coefficient studies. Fig 1 gives a typical arrangement for high pressure TEP studies on metallic samples. It would suffice here to mention that in the differential mode of TEP measurement it is required to keep the mean temperature of the sample constant with a provision to vary the temperature difference between the ends of the sample independently. The two heater arrangement with the electronic circuitry (both hardware and software) which will be described in the next section provides the control for both the mean temperature and temperature gradient. The complete details of the cell arrangement are described elsewhere. For measurements of TEP and resistivity at higher temperatures (upto 1000°C) the cell arrangement using a solid pressure transmitting medium and a graphite heater is employed.

3 Temperature programmer

The temperature controller / programmer is the heart of the high resolution TEP and resistivity measurement system. It essentially comprises of the following sub-
PC based data acquisition system

systems viz., signal amplifier with cold junction compensation, 16 bit A/D converter, a temperature linearising software, a software for simulation of the PID algorithm, a D/A converter and a power amplifier. All these sub-systems are controlled through a personal computer. Fig 2 gives the block diagram of the temperature programmer.

Figure 1: Teflon cell assembly with two heaters.

Figure 2: Block diagram of the temperature lineariser and controller.

The signal amplifier consists of an averaging circuit which averages the thermocouple voltages at the two ends of the sample followed by an instrumentation amplifier (Burr Brown 1NA101) configured for a gain of 200. It is clear that for temperature measurement using a single junction thermocouple, reference point compensation is
required. The temperature transducer AD592 (Analog Devices) which produces a voltage proportional to the temperature has been used as a cold junction compensator (CJC). The amplified thermocouple voltage with CJC corresponding to the mean temperature of the sample is then digitised using a precision 16 bit A/D converter. This digitised signal is then converted to a value corresponding to the temperature of the sample using a linearisation software. The software essentially fits a sixth order polynomial to the digitised thermo-emf E (in mV) through the relation

\[ T = a_0 + a_1 E + a_2 E^2 + \ldots + a_6 E^6 \]

where \( T \) is the linearised temperature. \( a_0 \ldots a_6 \) are constants for a given thermocouple. Since the pressure effect on the thermo-emf of chromel-alumel is small,\(^{14}\) this thermocouple offers distinct advantages in transport property measurements at high pressures. The fitting accuracy is \( 0.1^\circ C \) in the temperature range 0-1000\(^\circ C\).

The design of a temperature controller incorporating proportional, integral and derivative (PID) control in a feedback loop has been discussed by Forgan.\(^{13}\) It is worth mentioning that to achieve good temperature control, the positioning of the sample relative to the heater is crucial. The thermocouples, sample and the heater assembly should be in close thermal contact. Since teflon and the talc assembly are poor thermal conductors, the time constant of the heating block which essentially determines the rate of cooling is rather large favouring good temperature stability. We have developed a software to simulate the behaviour of an analog PID controller suitable for high pressure studies described elsewhere.\(^{12}\) A control signal is computed in real time which comprises of an error signal (difference between the set point and the actual temperature) with a proportional gain, \( K_P \), its integral and derivative terms with gains \( K_I \) and \( K_D \) respectively. The ease with which these gain factors can be changed facilitates fine tuning of these parameters for both the teflon and the high pressure - high temperature cells. The value of this control signal is then converted into a suitable analog signal using a D/A converter. This analog PID signal which is in the range 0-10V can be easily transformed to a power signal using a high voltage - high current operational amplifier. The power requirement for attaining temperatures up to 300\(^\circ C\) in the teflon cell using nichrome heater is in the range of 30V and 3A. This power signal can also be utilised to control the current on the primary side of a transformer that powers the graphite furnace in high temperature - high pressure cell. Fig 3 gives the step response of the controller in the teflon thermopower cell. For a
PC based data acquisition system

75°C step the time taken for stabilisation is of the order of 100 s. Fig 4 gives the temperature stability plot during a typical isothermal run. Temperature stability of the order of ± 0.02°C has been achieved in teflon cell while it is around ± 0.1°C in the high temperature cell assembly. This is nearly two orders of magnitude superior to that quoted by others in the field of high pressures. It is obvious that a temperature controller with sufficiently fast control action can be easily converted to a temperature programmer. If the set point value in the PID algorithm is increased linearly with respect to time, the temperature of the sample would follow suit provided the heating rate selected is slower than the rate of control action. Linear rate of heating/cooling which is software selectable from 1°C/minute to 20°C/minute makes this system useful for other techniques such as high pressure DTA and studies on non-equilibrium systems like glasses.
4 TEP measuring system

The absolute TEP, $S$, of the sample, in the differential mode of measurement, is given by the general expression1

$$S(T) = S_{Chr}(T) - S_{Chr-Alu}(T) \left[ \frac{V_{Chr-Sam-Chr}}{V_{Chr-Sam-Chr} - V_{Alu-Sam-Alu}} \right]$$  (1)

where $S_{Chr}(T)$ and $S_{Chr-Alu}(T)$ are the absolute TEP of chromel and the relative TEP of the chromel-alumel thermocouple respectively. $V_{Chr-Sam-Chr}$ and $V_{Alu-Sam-Alu}$ are the differential voltages developed across the thermocouples formed out of the reference probes and the specimen, when a small temperature difference is maintained across the length of the sample. $T$ is the mean temperature of the sample. It is clear from this equation that in order to evaluate $S$ as a function of mean temperature, it is necessary to simulate the temperature dependence of both $S_{Chr}$ and $S_{Chr-Alu}$.

Fig 5 gives the block diagram of the TEP measuring system. The design of the system is centered around the temperature controller described earlier. An important requisite for TEP measurement is the control of the temperature gradient. The control of the gradient should be such that, while the mean temperature is held constant, the magnitude of the temperature difference across the ends of the sample can be altered at will. Since TEP in the differential mode of measurement is related to the limiting value of $V_{Chr-Sam-Chr}/(V_{Chr-Sam-Chr}.V_{Alu-Sam-Alu})$ as the temperature difference tends to zero, the system should have the facility to evaluate this quantity for different temperature differences keeping $T$ constant. These requirements have been achieved by employing two separate temperature controllers. The mean temperature $T$ is one of the controlled variables while the temperature gradient can be varied independently by controlling the temperature of one of the ends of the sample by a separate controller.

The non-linear variations with temperature of the physical quantities like the absolute TEP of chromel ($S_{Chr}$) and the relative TEP of chromel-alumel thermocouple ($S_{Chr-Alu}$) are simulated by employing a sixth order polynomial curve fit using expressions of the type

$S(T)_{Chr-Alu} = b_0 + b_1T + b_2T^2 + ...+ b_6T^6$

$S(T)_{Chr} = c_0 + c_1T + c_2T^2 + ...+ c_6T^6$

where $b_0,...,b_6$, and $c_0,...,c_6$ are constants.
These constants can be evaluated from a computer fit of the NBS data. An algorithm based on the above expressions forms the basis for the two simulators $S_{\text{Chr-Alu}}$ and $S_{\text{Chr}}$ in the block diagram.

![Schematic block diagram of the TEP measuring system.](image)

Figs 6 and 7 present the simulated behaviour of these quantities as a function of temperature. In order to obtain a higher precision in the fit, the temperature range was divided into two blocks namely 0 - 200°C and 200 - 1000°C. The fitting error for
$S_{Chr-Alu}$ in the two ranges are $\pm 0.004 \, \mu V/{}^\circ C$ and $\pm 0.03 \, \mu V/{}^\circ C$. For $S_{Chr}$, the fitting errors are $\pm 0.001 \, \mu V/{}^\circ C$ and $\pm 0.1 \, \mu V/{}^\circ C$ in the two ranges.

![Figure 6: Polynomial curve fit of $S_{Chr-Alu}$ as a function of temperature. NBS data - *.
Solid curve is the fit.](image1.png)

![Figure 7: Performance of the $S_{Chr}$ simulator. NBS data - +. Solid curve is the fit.](image2.png)

5. AC Response

The AC response of $S_{Chr}$ is shown in Fig. 8 as a function of temperature. The AC response of $S_{Chr}$ is shown in Fig. 8 as a function of temperature. We have used a lock-in amplifier with constant current excitation (Alu-Sam) to perform these measurements.

Figure 6 shows a quadrature phase of the lock-in amplifier. The AC signal is out of phase with the reference signal by a constant angle, which is constant for the well-calibrated sample, which can be approximately 90° ($\approx 0.1$ rad). The AC response of the sample when the reference signal is in a fully phase with the AC signal minimizes the contribution of the DC component to the AC signal.

Similarly, the AC component of the signal is minimized when the AC signals are in quadrature.
the sample is held constant to within ± 0.02°C in the teflon cell and data collected as a function of pressure. The overall accuracy of TEP measurement is ≈ 0.5% and the resolution is around ± 0.01 μV/°C.

5 AC resistivity set up

The standard four-probe method of measuring resistivity as a function of temperature at high pressures generally requires six leads to be taken out from the high pressure cell of diameter less than 12 mm. This poses some experimental difficulties. We have developed a novel technique for AC resistivity measurement where only four leads are taken out from the high pressure cell.4

The two pairs of thermocouples attached to the sample are used for passing the AC current (across Chr-Sam-Chr), measuring the AC voltage developed across the sample (Alu-Sam-Alu), and also for measuring the temperature (DC voltage developed across the thermocouples).

Fig 8 gives the block diagram of the AC resistivity set up. The system consists of a quadrature oscillator, a high output impedance AC constant current source and a lock-in-amplifier coupled to a PC based DAS. The quadrature oscillator constructed out of two analog multipliers features two sine wave outputs at quadrature whose frequency can be varied from 10 Hz to 1 kHz. One of the sine wave outputs forms the reference signal for the lock-in-amplifier while the other output drives a constant current source. The design of the constant current source is centered around the well known Howland circuit. It would suffice here to mention that the current can be varied continuously from 0-50 mA and the high output impedance (≈ several megaohms) ensures that the current through the metallic sample is constant (≈ 0.01%) irrespective of its impedance. The AC voltage developed across the sample which is proportional to the sample resistance is measured with a lock-in-amplifier configured for selective external mode of operation. This ensures high sensitivity and full phase control. The frequency of the AC signal is chosen to be around 400 Hz to minimise the line frequency noise. The output voltage from the lock-in-amplifier is then digitised using a 16 bit A/D converter and after digital filtering is accessed in the PC.

Simultaneous measurement of temperature is achieved by measuring the DC component of the differential voltage developed across the current and voltage leads which are in close proximity. The contribution to the DC voltage from the small portion of
the sample between these two leads is negligible by the law of intermediate metals. A low pass active filter essentially removes the AC component and the DC output

![Diagram of AC resistivity setup](image)

**Figure 8: Block diagram of the AC resistivity setup.**

forms the input to the temperature lineariser and controller described earlier. It is thus possible to carry out high resolution AC resistivity studies either in the isobaric or isothermal mode of measurement. In this setup it is possible to detect changes of resistivity of the order of 1 part in a 1000.

6 Performance

The effect of subtle changes in resistivity due to induced currents and lattice anomalies is of considerable interest in nickel. Figures 9 and 10 demonstrate the sensitivity and commercial potential of this method. The results of experiments using this setup are reported in these results.

![Data for nickel](image)

**Figure 9: Data for nickel.**

![Graph for nickel](image)

**Figure 10: Graph for nickel.**
6 Performance

The PC based TEP and resistivity systems were mainly developed to study subtle changes in the electronic band structure accompanying several pressure induced continuous phase transformations. We have observed for the first time distinct anomalies in both TEP and resistivity near a pressure induced second order point in nickel. Further, high resolution TEP measurements have been used to track the commensurate-incommensurate phase boundary in a chromium-rhodium alloy. Fig 9 and 10 present some typical data on the resistivity and TEP behaviour across the magnetic transition in nickel at 20 kbar. The notable feature in these unprocessed experimental plots is the negligible scatter in the data. The detailed implications of these results will be published elsewhere.

Figure 9: Resistance as a function of temperature across the magnetic transition in nickel.

Figure 10: Isobar of TEP vs temperature across the Curie point.
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References

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16. T.G.Ramesh and V.Shubha, *(this volume).*