

# Studying Phase Transitions with a Strain Gage

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Many physical substances undergo phase transitions when subject to changes in environmental parameters. The transition of ice into water and water into steam are familiar examples. Similarly, chromium undergoes a phase transition at a specific temperature, called the Néel temperature changing its magnetic order from the antiferromagnet to the paramagnet state. This magnetic reordering is accompanied by a change in volume. In other words, the phase transition is a magnetoelastic phenomenon. In this experiment, we study the phase transition of chromium by directly measuring the volume change. Several important thermodynamical concepts will also be navigated.

## KEYWORDS

Antiferromagnetism · Paramagnetism · Néel Temperature · Strain Gage · Latent Heat · Gibbs Function · Wheatstone Bridge · Instrumentation Amplifier ·

**APPROXIMATE PERFORMANCE TIME** 1 week.

## 1 Objectives

The objectives of the present experiment are to,

1. understand the meaning of phase transitions and the order of transitions,
2. how change in volume occurs as the temperature changes and especially at the phase transition,
3. understand the relationship between the paramagnetism and antiferromagnetism, magnetism and thermodynamics, and thermodynamics and elastic properties of materials,
4. learn the use of the strain gage,
5. understand the purpose and significance of the instrumentation amplifier and the bridge circuitry which are general-purpose measured techniques.

# References and Essential Reading

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## 2 Introduction

Chromium acts as an antiferromagnet below the Néel temperature but at the Néel temperature ( $T_N \approx 38^\circ\text{C}$ ), it undergoes a phase transition and becomes paramagnetic. This phase transition occurs due to the change in the orientation of magnetic dipoles as shown in Fig 1. Thermodynamically this magnetic transition is of first order. We can easily detect this transition by measuring the volume change  $\Delta V$  in chromium.

In antiferromagnetic materials such as chromium, the interaction between the magnetic moments tends to align adjacent moments antiparallel to each other as illustrated in Fig. 1. Although one set of magnetic ions is spontaneously magnetized

below some critical temperature (called the Néel Temperature,  $T_N$ ), the second set is spontaneously magnetized by an equal amount in the opposite direction. As a result, antiferromagnets have no net spontaneous magnetization. Excellent resources on magnetism in materials are the references [1] and the more detailed [2].

Antiferromagnets are being widely used in many applications such as GMR sensors, spin valves, MJT's and computer hard disks where antiferromagnets are coupled to ferromagnets through the exchange interaction. Antiferromagnetic materials occur commonly among transition metal compounds, common examples include heavy-fermion superconductor URu<sub>2</sub>Si<sub>2</sub>, alloys such as iron manganese (FeMn), oxides such as nickel oxide (NiO) and the most popular example, manganese oxide (MnO).

**Q 1.** Why do we prefer measuring the volume change at the Néel transition instead of directly detecting the magnetic reordering?

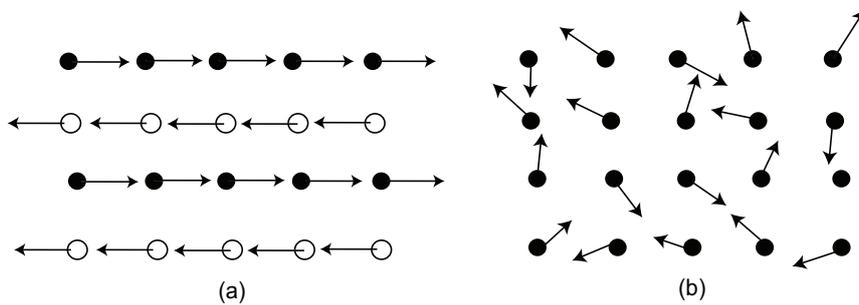


Figure 1: Phase transition of chromium from (a) antiferromagnetic to (b) paramagnetic phase at  $T_N$ . In the antiferromagnetic lattice, neighboring spins are anti-aligned. The antiferromagnetic state can be represented by two ferromagnetic sublattices  $A$  and  $B$  which are aligned antiparallel to each other. These sublattices are represented by filled and empty circles in (a).

## 2.1 First Order Phase Transition

In thermodynamics [3], the change of state such as ice melting to water or water vaporizing into steam are referred to as transitions of phase. This transition in phase accompanies change in entropy  $S$  and volume  $V$  while the pressure  $P$  and temperature  $T$  remain constant. The first order phase transition can be defined by considering the Gibb's function  $G(P, T)$ . Note that the natural variables for  $G$  are  $P$  and  $T$  and is defined through the Legendre transform,

$$G = H - TS. \quad (1)$$

If the process is reversible and infinitesimal, we have the characteristic equation,

$$dG = VdP - SdT. \quad (2)$$

**Q 2.** Derive Equation (2) from (1) and the first law of thermodynamics.

Since  $dG$  is an exact differential and the variables  $P$  and  $T$  are independent, one can write the molar entropy and volume as differentials of the Gibbs's energy,

$$S = - \left( \frac{\partial G}{\partial T} \right)_P \quad \text{and} \quad (3)$$

$$V = \left( \frac{\partial G}{\partial P} \right)_T. \quad (4)$$

Considering these relationships, we say that the phase transition is of first order only if it satisfies the following equivalent requirements.

1. The molar entropy and molar volume change at the phase transition.
2. The first-order derivatives of the Gibbs function with respect to temperature and pressure change discontinuously. This predicted behavior is shown in Fig. 2.

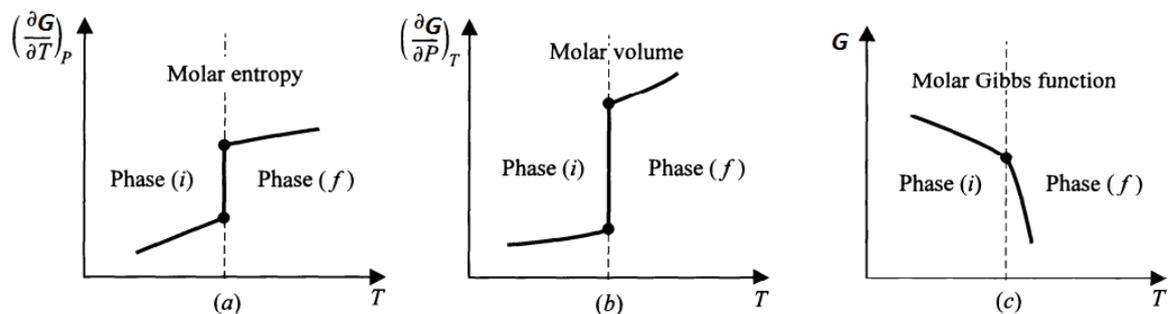


Figure 2: Characteristics of a first-order phase transition. There are discontinuous changes in (a) molar entropy and (b) molar volume whereas the (c) Gibbs function is single valued with a discontinuous slope.

**Q 3.** What is a second order phase transition?

**Q 4.** Enumerate common first and second order phase transitions.

**Q 5.** What is the physical meaning and significance of Gibbs free energy?

Volume is a directly observable parameter, whereas entropy is impossible to measure directly. In the current experiment, we will monitor the change in volume that occurs in Cr as its temperature is varied expecting a discontinuous change at the Néel temperature.

## 2.2 Clausius-Clapeyron Equation

Phase transitions are generally understood with the help of phase diagrams that constitute powerful tools in thermodynamics and materials science. A phase diagram characterizes a discontinuous phase transition between two phases on a pressure–temperature ( $PT$ ) graph. Consider the example of phase transition of

ice melting into water and then water into vapors, this transition is depicted in the  $PT$  diagram of Figure 3. The line separating two phases is known as a co-existence curve. Along the coexistence curve,  $dG = 0$  or  $G = \text{constant}$ . The Clausius-Clapeyron equation [3] gives the slope of this coexisting curve,  $dP/dT$  and is applicable to first-order phase transitions, for which  $dP = dT = 0$  along the phase transition. The equation is,

$$\frac{dP}{dT} = \frac{\Delta H}{T\Delta V} = \frac{L}{T\Delta V}. \quad (5)$$

where,  $L$  is the latent heat involved in the isobaric phase transition,  $\Delta V$  is the change in volume during the phase transition, and  $\Delta H = L$  is the difference in enthalpies of the final and initial phases.

**Q 6.** Derive the Clausius-Clapeyron equation.

**Q 7.** What does the black dot in Figure 3 signify?

**Q 8.** For the melting of ice into water, the slope of the melting coexistence curve is negative (as seen in Figure 3). Using this fact, argue why the melting point increases as the atmospheric pressure decreases. Why does water contract on melting? Why do glaciers float on water and what causes the glaciers to melt even below the normal melting point of ice?

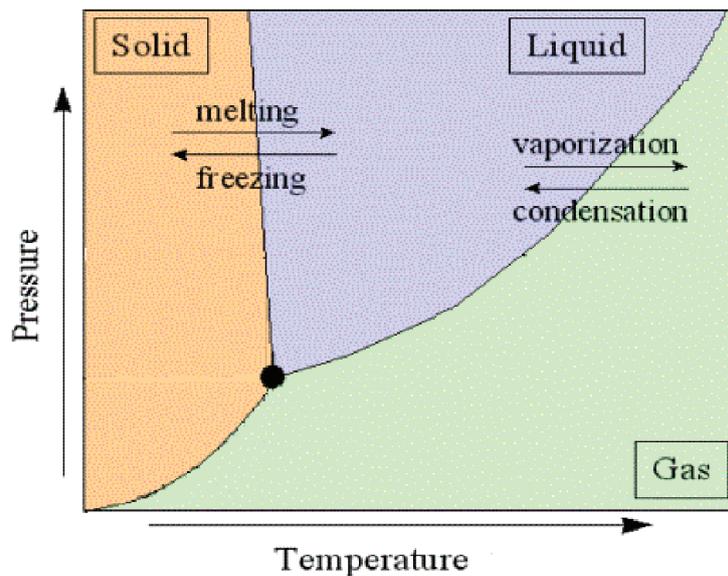


Figure 3: Phase Diagram of water reproduced from [8].

### 3 Overview of the Experiment

A thermocouple and a strain gage have been attached to a piece of polycrystalline chromium metal as shown in Figure 4. The chromium metal is heated using a peltier heater and the output voltage of the instrumentation amplifier connected to a bridge circuit incorporating a strain gage, is plotted against temperature.

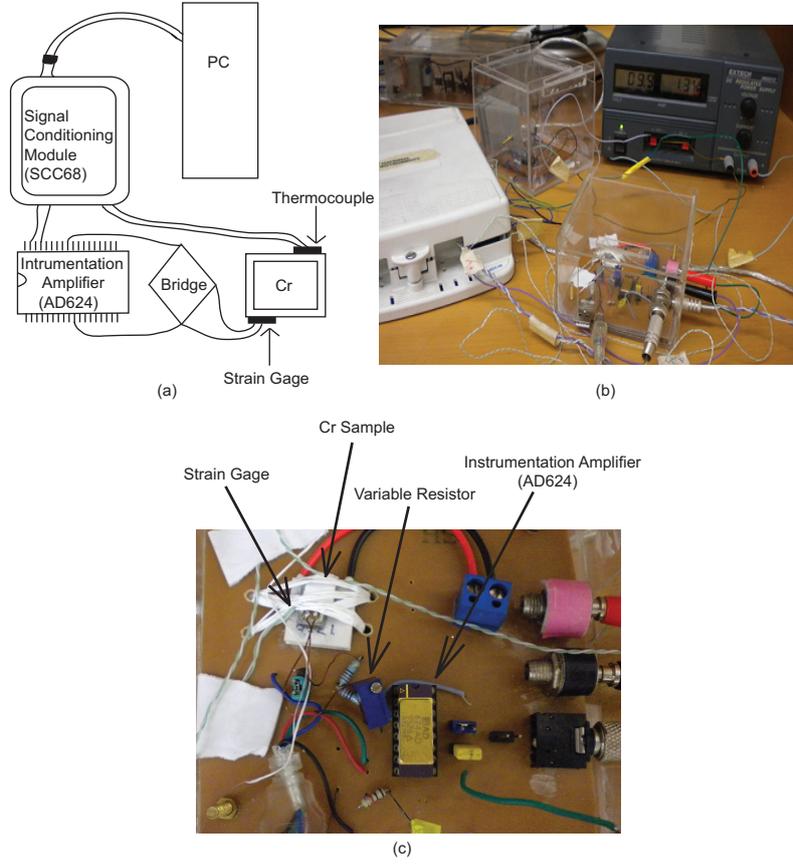


Figure 4: Experimental Setup.

### 3.1 Volume Measurement with the strain gage

When chromium undergoes a phase transition at Néel temperature, not only is its magnetic order transformed, there is also a change in its volume. We can measure this change with the help of a strain gage and some useful electronics. The thermally induced volume expansivity is characterized by the variable,

$$\beta = \frac{1}{V} \left( \frac{\partial V}{\partial T} \right)_P, \quad (6)$$

and using the characteristic equation (2) as well as the Maxwell's relations, we can derive,

$$T dS = C_p dT - V \beta dP, \quad (7)$$

$C_p = T(\partial S/\partial T)_P$  being the heat capacity at constant pressure. For a phase transition, both  $dT$  and  $dP$  are zero, but since the heat transferred during the phase transition  $T dS$  is finite, we have  $C_p \rightarrow \infty$  and  $\beta \rightarrow \infty$ , indicating a discontinuous change in the volume *at* the phase transition.

The strain gage is directly glued to the metal with the help of specialized adhesives and bonding pads [5, 6]. The fractional change in the resistance of strain gage is related to the fractional volume change in the polycrystalline chromium. The volume expansivity  $\beta$  and the linear expansivity (of any material) are related through the simple relationship,

$$\beta = \frac{3}{\Delta T} \frac{\Delta l}{l}. \quad (8)$$

**Q 9.** Verify the relationship (8), which is only true for a cubic single crystal or polycrystalline material with randomly oriented crystallites.

**Q 10.** Derive equation (7).

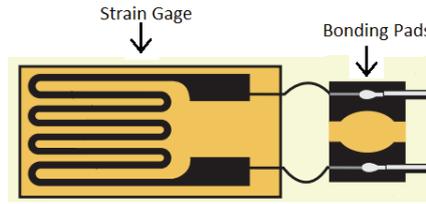


Figure 5: Linear strain gage, with bonding pads. Connective wires are soldered to the bonding pads instead of being directly attached to the gage wire.

The strain gage, for example the one shown in Figure 5, is a device used to measure the strain  $\Delta l/l$  of an object to which it is glued. The output resistance of the strain gage is a function of strain and can be interrelated with the help of a gage factor  $F$ ,

$$F = \frac{\Delta R/R}{\Delta l/l}. \quad (9)$$

In this experiment we are using the LY11 strain gage from Omega Engineering [6]. Table 1 provides some important characteristics of LY11 supplied by the manufacturer.

Gage Factor	2
Temperature compensation	Steel
Nominal resistance at room temperature	$120 \Omega \pm 1\%$
Active gage length at room temperature	$1.50 \pm 0.059 \text{ mm}$
Max voltage drop allowed	$2.5 V_{rms}$

**Table 1.** Characteristics of Omega’s LY11 strain gage. See the website [6] for more details.

### 3.2 Peltier Heater

When two wires of dissimilar conductors or semiconductors are combined to form a junction and an electric current is passed through these wires, a temperature difference is developed across the junction. One side of the junction will be hot and the other will be cold, depending on the temperature difference. This effect of developing a temperature difference is called the peltier effect, discovered by French physicist Jean Charles Peltier in 1834. One side of the junction might be hot and the other might be cold, depending on the temperature difference. Thermoelectric module also called peltier heaters consists of an array of p- and n-type semiconductor elements and uses the same peltier effect to produce heat.

### 3.3 Temperature measurement with the thermocouple

The thermocouple is a transducer that converts temperature into voltage. By now, you will be familiar with its operation. In the present experiment we using a  $J$ -type thermocouple. The thermocouple has been pre-calibrated and is directly connected to the Data Acquisition (DAQ) board which has a built-in thermistor for cold junction compensation. Table 2 provides some important characteristics of the  $J$ -type thermocouple. You are recommended to consult the strain gauge, thermocouple and peltier heater data sheets which also are available on the Physlab website.

Temperature Range	-40 to +750 °C
Combination of alloys	chromel and constantan
Sensitivity	Approx. 55 $\mu$ V/°C

**Table 2.** Some relevant characteristics of the  $J$ -Type thermocouple.

### 3.4 Coefficient of Thermal Expansion

Since the strain gage is made of steel and has its own thermal expansion, so when the strain gage is bonded to the object (in our case chromium), the strain gage bears thermally induced apparent strain per degree change in temperature. The thermally induced strain per unit temperature change is given by the equation,

$$\frac{\Delta l/l}{\Delta T} = \frac{\alpha}{F} + |\beta_s - \beta_c|, \quad (10)$$

where,  $\alpha$  is the temperature coefficient of resistance of the strain gage and for steel its value is  $6.25 \times 10^{-10} \text{ K}^{-1}$  [13],  $F$  is the gage factor of the strain gage,  $\beta_s$  is the coefficient of thermal expansion of steel strain gage, and  $\beta_c$  the coefficient of thermal expansion of chromium. The thermal expansivity of steel can be looked up from reference tables such as [10] or the CRC Handbook.

Some specialized electronics is required to measure the strain with a strain gage. In this experiment we use a Wheatstone bridge and an instrumentation amplifier to directly measure the change in resistance induced by the change in volume.

### 3.5 Wheatstone Bridge

The Wheatstone bridge is normally used to measure an unknown resistance when placed in the bridge configuration, the arrangement of a typical bridge is shown in Figure 6.

In our circuit  $R = 120 \Omega$ ,  $R_g$  is the resistance of the strain gage and  $R_{\text{adj}}$  is the variable resistance used to balance the bridge. The  $R_{\text{adj}}$  is adjusted such that the voltage reading at the output of the bridge is zero ( $V_g = 0$ ). We can then use the

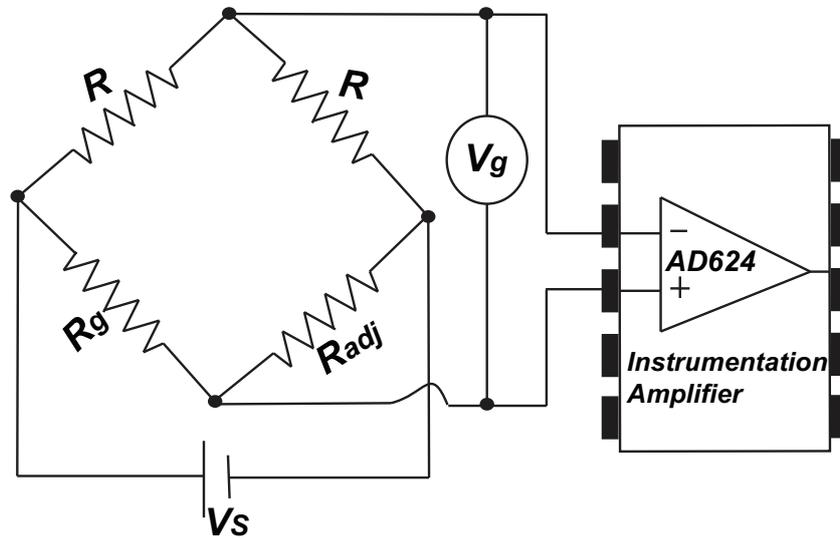


Figure 6: Wheatstone bridge configuration for measuring  $R_g$ .

bridge equation to evaluate the change in resistance  $R_g$ ,

$$V_g = \left( \frac{R_g}{R + R_g} - \frac{1}{2} \right) V_s \quad (11)$$

where,  $V_s \approx 1.5$  V, is the supply voltage and  $V_g$  is the output voltage.

**Q 11.** Derive equation (8) from first principles of circuit analysis.

### 3.6 Instrumentation Amplifier

An instrumentation amplifier is used when high and stable gain is required, especially in the amplification of weak signals, say, in the order of microvolts. Ordinary amplifiers have very low noise rejection ability and hence when they are fed in with very small input signals, the output signal gets distorted. For the same reason the instrumentation amplifier is the most preferred device in applications involving weak signal measurements such as ECG and EEG, strain gage systems to detect cracks in masonry as well as precise resistance thermometers.

In the current experiment, we use the AD624 instrumentation amplifier [9]. The block diagram of AD624 is shown in figure 7. It can be noted that the instrumentation amplifier is a combination of ordinary amplifiers with some precise matching resistances. **We use AD624 with the gain value configured at 1000.**

## 4 Experimental Procedure

1. Turn on the power supply and make sure the voltage is set to 1.3 Volts.
2. Open and run the labview file titled **Cr-phase-study.vi**. Set the **Start-Heating** control to 1 and **Max-Temperature** to 45. The programme will start acquiring and saving data from the thermocouple and the strain gage. The output graphs and other details on the front end are self-explanatory.

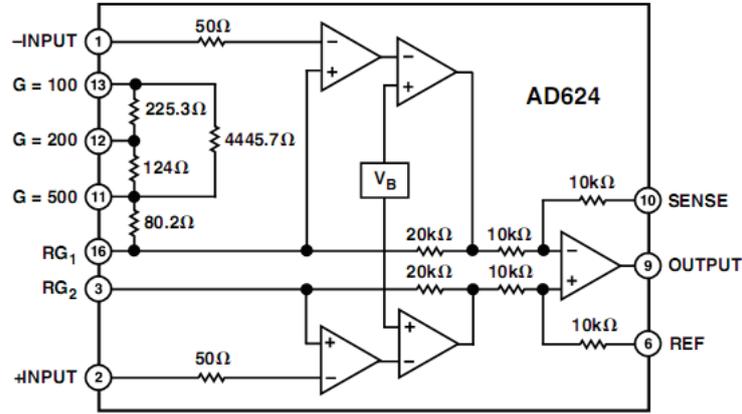


Figure 7: Block Diagram of the instrumentation amplifier AD624.

3. Observe the temperature reading. It should be around room temperature.
4. Again observe the temperature reading which is continuously rising as the sample is heated.
5. The sample will continue to heat up till the temperature reaches  $45^{\circ}\text{C}$ , at that point the heater will stop and the sample will start to cool down.
6. The labview file will stop as the sample is cooled down to  $30^{\circ}\text{C}$ .
7. Plot  $V$  (instrumentation amplifier output voltage) against temperature  $T$  using the Matlab file **Exp-graphs.m**.

## 5 Experimental Objectives

1. By identifying the transition in the  $V$ - $T$  plot, estimate the Néel Temperature  $T_N$ .
2. What is the estimated uncertainty or standard error in  $T_N$ ?
3. Note down the dip in the value of  $V_g$  at  $T_N$ .
4. Find the change in resistance ( $\Delta R$ ) of the strain gage with the help of the bridge equation. *Note: Before using the bridge equation compensate the value of  $V_g$  with the AD624 gain  $G$ .*
5. What is the uncertainty in the value of  $\Delta R$ ?
6. Calculate the value of the latent heat ( $L$ ) per unit volume associated with the phase transition at Néel Temperature. Use the Clausius-Clapeyron relation, with,  $dT/dP = -1.3 \times 10^{-7}$  K/Pa [4]. The chromium sample used in this experiment has a mass of 1.3 g. Compare your latent heat with values published in [12].
7. How has the uncertainty propagated from  $\Delta R$  into  $L$ ? Show the complete steps and calculations.

8. Calculate the change in entropy at the Néel temperature.
9. Determine the values of  $\beta_c$  (the coefficient of thermal expansion of chromium) above and below the Néel temperature. What is the uncertainty in the value of  $\beta_c$ ? Do not lose sight of the thermally induced strain of the strain gauge (10), that must be accounted for in your calculations.
10. For  $T < T_N$ , is  $\beta_c$  positive or negative? If solid Cr is subject to external pressure, how will the entropy react? Will the Cr atoms become more or less orderly as the pressure is isothermally increased? HINT: Derive the Maxwell relation corresponding to the characteristic function  $dG = -SdT + VdP$ .