APPARATUS AND DEMONSTRATION NOTES

Jeffrey S. Dunham, *Editor*
Department of Physics, Middlebury College, Middlebury, Vermont 05753

This department welcomes brief communications reporting new demonstrations, laboratory equipment, techniques, or materials of interest to teachers of physics. Notes on new applications of older apparatus, measurements supplementing data supplied by manufacturers, information which, while not new, is not generally known, procurement information, and news about apparatus under development may be suitable for publication in this section. Neither the *American Journal of Physics* nor the Editors assume responsibility for the correctness of the information presented. Submit materials to Jeffrey S. Dunham, *Editor.*

A classroom experiment to demonstrate ferroelectric hysteresis

M. Dawber, I. Farnan, and J. F. Scott
Earth Sciences Department, Downing Street, University of Cambridge, Cambridge CB2 3EQ,
United Kingdom

(Received 19 July 2002; accepted 24 January 2003)

We have developed a classroom experiment suitable for undergraduate students in which they fabricate a ferroelectric capacitor from potassium nitrate and then observe the electrical behavior as the film is cooled through its transition temperature. The experiment can be carried out using a capacitance bridge that is simple to construct and inexpensive. The experiment gives students a hands-on experience with ferroelectric phenomena, a subject of considerable interest from both a fundamental and a technological standpoint.
[DOI: 10.1119/1.1561271]

I. INTRODUCTION

There are at least three systems in nature that exhibit hysteretic responses to driving forces: ferromagnets, ferroelectrics, and ferroelastics. (The latter are materials that have hysteresis in their stress–strain relationships.[1]) Of the three systems, only ferromagnets are usually the subject of undergraduate laboratory experiments. A ferroelectric is a material in which an electric dipole moment is present even in the absence of an external field. This typically occurs as a result of a change in the crystal structure so that the centers of positive and negative charge in the crystal do not coincide. A brief section on ferroelectric phase transitions is included in Kittel's textbook[2] and more detailed information can be obtained from Fatuzzo and Merz.[3] Normally it is difficult to demonstrate ferroelectricity in the classroom laboratory as the coercive fields of most materials are of the order of kV/cm, making experiments on bulk ferroelectrics potentially dangerous and unsuitable for students. Ferroelectrics in thin film form, on the other hand, may be switched with a few volts and there is now both production of and research on nonvolatile memories based on ferroelectric thin films.[4] Typically, however, thin films are deposited in expensive chemical vapor deposition or pulsed-laser deposition machines, making the fabrication of thin films by students as part of a teaching experiment impractical. We present here an experiment in which students make and characterize a ferroelectric thin film capacitor. The method outlined here is both safe and inexpensive.

Potassium nitrate is a material with a ferroelectric phase (phase III) which in bulk form is stable only between 115 and 125 °C, but is metastable at room temperature. It has been found that when KNO_3 is made as a thin film, the lower temperature limit of the ferroelectric phase is lowered, and it can be stable at room temperature.[5] Phase diagrams for bulk KNO_3[6] and for thin films[7] are shown in Figs. 1 and 2, respectively. In bulk the phase transition is a re-entrant phase transition, i.e., it can be reached only on cooling and not by heating. This is because the ferroelectric phase is narrower than the thermal hysteresis.

Potassium nitrate is not used for commercial applications because the challenges of fabricating capacitors have proven too difficult. KNO_3 readily absorbs water from the atmosphere, thereby severely degrading its ferroelectric properties. The main materials used today for ferroelectric memories are lead zirconate titanate and strontium bismuth tantalate. Because potassium nitrate melts at about 330 °C it is quite straightforward to melt the powder and then cool it to form a thin film in which hysteresis properties may be measured with an acceptably low voltage. We have designed an experiment to fabricate and measure the properties of a KNO_3 capacitor that is suitable for an undergraduate course. This experiment was run successfully this year as part of the first year course ‘‘Materials and Mineral Sciences 1A,’’ a shared course between the Department of Earth Sciences and the Department of Material Sciences at the University of Cambridge.

II. SAWYER-TOWER CIRCUIT

The standard circuit used to measure a ferroelectric hysteresis loop is the Sawyer-Tower circuit.[8] Our implementation of this circuit is shown in Fig. 3.

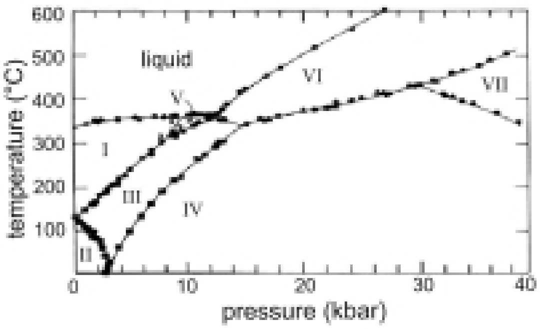


Fig. 1. Phase diagram for bulk KNO_3 with temperature as a function of hydrostatic pressure.

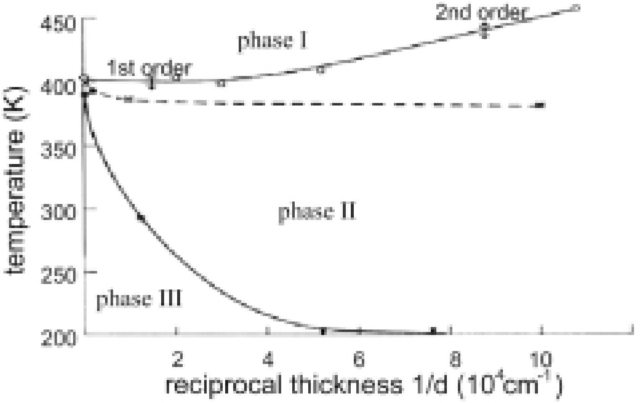


Fig. 2. Phase diagram for KNO_3 thin films with temperature as a function of reciprocal thickness.

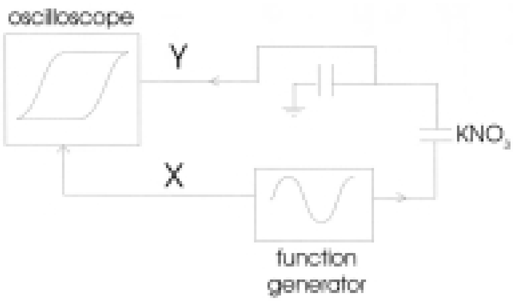


Fig. 3. Sawyer-Tower circuit as implemented in the experimental setup described here.

By measuring the potential V across a standard capacitor in series with the KNO_3 film one can determine the charge Q on the KNO_3 capacitor using $Q = CV$. When two capacitors are in series the charge on each capacitor must be the same (in an ideal capacitive circuit no current flows), so the electric charges on the standard capacitor and the KNO_3 capacitor are the same. As the capacitance C of the standard capacitor is known we are able to calculate Q from the magnitude of the voltage signal we measure at the standard capacitor.

We display the signal applied to the material as the X signal of an X-Y trace on an oscilloscope. Most of the voltage drops across the KNO_3 capacitor because we have selected a high value of capacitance for the standard capacitor, so we can consider the X signal to represent the voltage across the sample. The Y signal is proportional to the charge on the KNO_3 capacitor.

If this technique were used on an ordinary linear dielectric one would expect a linear response (a straight line on the x-y display), as the polarization is directly proportional to the field applied. In practice there is some opening of the loop, which is due to dielectric loss. In a ferroelectric there is a remnant polarization, i.e., the polarization charge remains aligned in the direction it was poled by the applied field even after this field has been removed. The electrical polarization characteristics of the capacitor thus depend on the history of the field that is applied to it and hence it displays hysteresis.

III. NECESSARY EQUIPMENT

The following equipment is necessary for this experiment:

A. Electrical equipment

(1) A basic oscilloscope. It must be capable of operating in X-Y mode. It only needs to be capable of operating at low frequencies (100–1000 Hz).

(2) A signal generator. It should be capable of producing sine waves of 15-V amplitude. Frequencies in the range of 100–1000 Hz give the best hysteresis measurements.

(3) Hotplate. It should be capable of heating the sample to at least 350 °C. It should be possible to clamp the metal sheet to the edge of the hot plate.

(4) Thermocouple. A thermocouple is used to measure the temperature of the capacitor during the experiment.

(5) Test box. A photograph of the test box is shown in Fig. 4. The total cost of the components for this box is very low and it can be assembled easily. The audio transformer

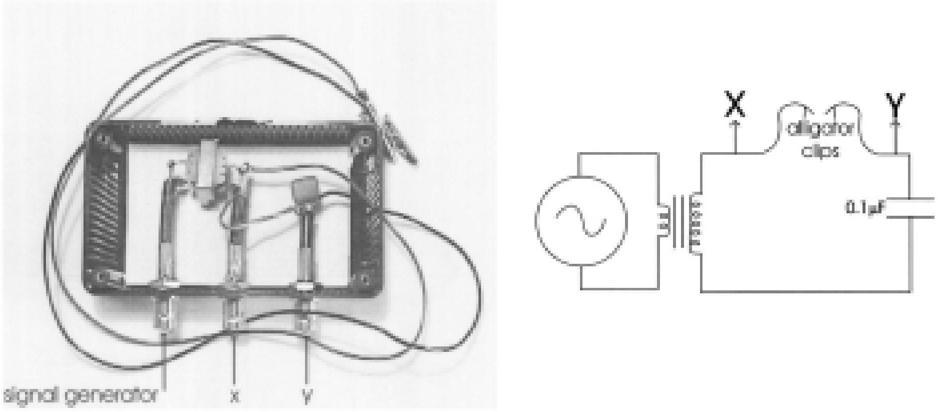


Fig. 4. The test box for the hysteresis measurement. On the left is displayed the actual components; on the right is a diagrammatic representation of the circuit.

Fig. 5. Assembled experiment before heating. The metal sheet is clamped to the heater between glass slides. The thermocouple is placed below the top slide in contact with the metal sheet. KNO_3 powder is then distributed onto the metal sheet, and thumbtacks are placed on top of the powder.

simply provides more switching voltage. At high frequencies, the signal will become distorted; at the frequencies used in this experiment there is no distortion and this provides an economical way of providing a sufficiently large switching signal. The currents required are tiny. The rest of the circuit is the implementation of the Sawyer-Tower circuit shown in Fig. 3. There are two outputs from the test box. One is the input signal generated by the signal generator after amplification. The other is the signal across the standard capacitor. These are connected to the X and Y channels of the oscilloscope, respectively.

B. Other equipment

In addition to the electrical equipment outlined above, several other items are needed to run the experiment. KNO_3 powder, metal sheeting, e.g., aluminum and copper cut into small squares, and thumbtacks are all used in the experiment as consumables. Students will also require insulated gloves, glass microscope slides, and bulldog clips.

IV. EXPERIMENTAL METHOD

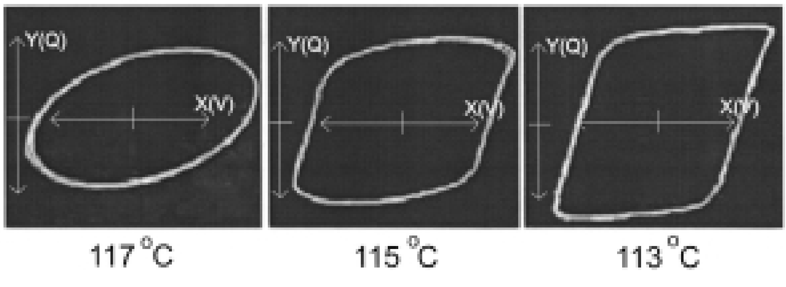
KNO_3 is highly hydrophilic and should be dried before use as the ferroelectricity is degraded when the material has absorbed water. We found that the easiest way to do this was to heat it in a conventional microwave oven for about 2 min. Care must be taken that the powder does not melt in the microwave, as in the molten state KNO_3 is conductive and arcing will occur. After drying, the powder can be stored in a desiccator until needed. KNO_3 is a powerful oxidizing agent, and although the risks of a dangerous reaction are small (we experienced no problems in any of our classes), the KNO_3 should be kept as pure as possible so as to avoid any reactions. In particular it should not be mixed with any carbon-based compound or placed near flames.

As a bottom electrode we used thin aluminum sheeting cut into a square of approximately 25 cm^2; a small tab on one corner was bent up to allow easy electrical contact with an alligator clip.

The films are made and measured in the same setup. A hotplate capable of heating to around 350 °C is required. Use of a naked flame for heating, such as that from a Bunsen burner, is not recommended because of the potential for ignition of the KNO_3. The aluminum sheet should be clamped to the hotplate, but must be electrically insulated from it. For this purpose we used bulldog clips and microscope slides. The plate was clamped between glass slides on top and bottom, leaving most of the top surface of the slide exposed. A thermocouple is inserted under the top slide. Once the experiment has been set up as described above a thin covering of potassium nitrate powder should be placed on the bottom electrode. About six to eight thumbtacks should be placed on the powder. We found that upon melting of the KNO_3, thumbtacks would automatically form a capacitor of suitable thickness; they are also easy to connect to the test box with an alligator clip. The setup of the experiment at this point is shown in Fig. 5.

Once the experiment has been set up the heater should be turned on until the KNO_3 is completely molten. At this point the heater may be turned off and the sample allowed to cool. At about 170 °C (above this temperature there is a risk that solder on the clips might melt!) the alligator clips may be attached, one to the bottom electrode and the other to any of the thumbtacks. It is not important which clip is attached to which contact. If at this point a signal is applied, the result on the oscilloscope will be a loop, essentially a linear response opened up by dielectric loss. It is best to apply a 25-V amplitude signal, (full-scale on the X axis for most oscilloscopes) as the coercive voltage for these capacitors is about 17–20 V depending on sample thickness. As the capacitor goes through the phase transition, the loop will change dramatically and become very square. Usually the scale of the Y signal on the oscilloscope needs to be changed. Figure 6 shows a typical result.

Various investigations can be carried out by students. They can easily vary the measurement frequency and assess its effect on remnant polarization and coercive field. The behavior as the temperature continues to fall may also be studied. As the temperature decreases the size of the signal will decrease. We found that 50(60) Hz noise frequently became significant at low signal amplitude. This gives the interested student an additional point of investigation. By selecting applied signal frequencies such as 50(60) and 100(120) Hz, a



117 °C 115 °C 113 °C

Fig. 6. Typical results seen on the oscilloscope display during the experiment that show the ferroelectric phase transition. The Y signal on the oscilloscope corresponds to the charge on the capacitor Q, while the X signal corresponds to the applied voltage V. Note that for the third picture (113 °C) the Y scale on the oscilloscope has been increased as the signal substantially increases when the capacitor undergoes the phase transition, i.e., the charge on the capacitor, Q, increases dramatically after the phase transition occurs.

student can see interference effects between in-phase noise and signal. Many of our students found this the most interesting part of the experiment.

V. CONCLUSIONS

We have developed an experiment to demonstrate ferroelectric polarization hysteresis to undergraduate students. The experiment is simple, safe, and inexpensive. We believe it would complement physics lectures on ferroelectricity or phase transitions in general. It may also be of interest to students studying electrical engineering, materials science, or mineral science. Students get hands-on experience with making and characterizing a ferroelectric capacitor. The feedback from students in our course was extremely positive.

[1] K. Aizu, ‘‘Possible Species of Ferromagnetic, Ferroelectric, and Ferroelastic Crystals,’’ Phys. Rev. B **2** (3), 754–B772 (1970).
[2] C. Kittel, *Introduction to Solid State Physics* (Wiley, New York, 1996), 7th ed., Chap. 13.
[3] E. Fatuzzo and W. J. Merz, *Ferroelectricity* (North Holland, Amsterdam, 1967).
[4] J. F. Scott, *Ferroelectric Memories* (Springer-Verlag, Berlin, 2000).
[5] J. P. Nolta and N. W. Schubring, ‘‘Ferroelectricity in Potassium Nitrate at Room Temperature,’’ Phys. Rev. Lett. **9** (7), 285–286 (1962).
[6] E. Rapoport and C. G. Kennedy, J. Phys. Chem. Solids **26**, 1995 (1965).
[7] J. F. Scott *et al.*, ‘‘Properties of ceramic KNO_3 thin-film memories,’’ Physica B **150**, 160–167 (1988).
[8] C. B. Sawyer and C. H. Tower, ‘‘Rochelle Salt as a Dielectric,’’ Phys. Rev. **35**, 269–273 (1930).

Verification of Bohr’s frequency condition and Moseley’s law: An undergraduate laboratory experiment

S. B. Gudennavar, N. M. Badiger,[a] S. R. Thontadarya, and B. Hanumaiah[b]
Department of Physics, Karnatak University, Dharwad-580 003, Karnataka, India

(Received 30 August 2002; accepted 28 February 2003)

We describe an undergraduate laboratory experiment to verify Bohr’s frequency condition and Moseley’s law using a thin NaI(Tl) detector spectrometer and a weak ^{57}Co source. The slope of the plot of $K\alpha$ x-ray energy versus $(Z-1)^2$ yields a value for the Rydberg constant, $R=(1.19\pm 0.01)\times 10^7\ \mathrm{m}^{-1}$, which is in fair agreement with the best literature value, $R=10\,973\,731.534(13)\ \mathrm{m}^{-1}$.
[DOI: 10.1119/1.1568969]

I. INTRODUCTION

Several student laboratory experiments on Moseley’s law have been described in AJP.[1–5] All of these experiments, although they are intended for the undergraduate level, involve sophisticated equipment, such as an x-ray machine, an x-ray diffractometer, an electron microscope, Ge(Li) or Si(Li) spectrometers, and intense radioactive sources. Not all undergraduate laboratories can obtain such equipment and also procure radioactive sources with activities of the order of 10^7 Bq. Even if strong sources are procured, they require heavy shielding to protect personnel from radiation. Since Moseley’s law should be seen by students at the undergraduate level, it would be desirable to make the experimental arrangement more practicable. In this direction, we have developed a low-cost arrangement that should suit any undergraduate laboratory, including those in underdeveloped countries.

Bohr’s theory of atomic structure successfully explains many experimental facts, including the emission of sharp spectral lines. According to this theory, the energy of an electron in its orbit, E_n, is given by

$$E_n=-\frac{R_\infty Z^2}{n^2}, \tag{1}$$

where R_∞ is the Rydberg energy for an infinitely heavy nucleus, Z is the nuclear charge, and $n=1,2,3,...$ is the principal quantum number used to designate energy levels. The emission of radiation from the atom, according to Bohr, is due to the transition of the atom from an initial higher energy state (E_i) to a final lower energy state (E_f), and the frequency ν of the emitted radiation is given by the condition $E_i-E_f=h\nu$, where h is Planck’s constant. Thus, for instance, $K\alpha$ x-ray emission is due to transfer of an L-shell electron ($n_i=2$, energy E_L) to the K-shell ($n_f=1$, energy E_K), where a vacancy has been created by some means (such as by irradiating the atom with γ rays) prior to the transition. Hence the energy of $K\alpha$ x ray is given by

$$E_{K\alpha}=E_L-E_K=\left(-\frac{R_\infty Z^2}{4}\right)-\left(-\frac{R_\infty Z^2}{1}\right)=\frac{3R_\infty Z^2}{4}, \tag{2}$$

which shows that the energy of characteristic x rays is proportional to square of the nuclear charge. In the x-ray notation, the subscript α refers to those transitions of electrons for which $\Delta l=\pm 1$, that is, for electric dipole radiations.

Moseley, who was studying $K\alpha$ x-ray spectra at the same time and in the same laboratory (Rutherford’s Manchester Laboratory, Manchester), as Bohr, used this expression, but modified Z to $(Z-1)$ to fit his experimental data. Thus, Moseley used

$$E_{K\alpha}=\frac{3R_\infty (Z-1)^2}{4} \tag{3}$$

to describe his data. Equation (3) is usually referred to as Moseley’s law.

In later years, the modification of Z to $(Z-1)$ or $(Z-S)$ was understood in terms of shielding or screening of nuclear charge by the surrounding electrons. So, in the original Moseley’s law, the factor $(Z-1)$ means the effective screening for the $K\alpha$ transition is unity.[6] Thus, from Eqs. (2) and (3) it is clear that various ideas about atomic structure and