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PCT-2-77/R3-79

November 16, 1979

SECOND ANNUAL REPORT

DEMONSTRATION OF SCIENTIFIC
AND ECONOMIC FEASIBILITY OF A
SOLID-STATE HEAT ENGINE

MASTER

DOE Contract No. ER-78-C-03-1736

Submitted to:
DEPARTMENT OF ENERGY
Washington, D.C. 20545

on

November 16, 1979

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I. INTRODUCTION

PCT is pleased to report the production of reproducible, controlled thermal cycles. These cycles were obtained with a new type of experimental heat engine. The temperature measuring and heat pumping approaches of the new experiment differed from the earlier vapor engine (whose initial thermal cycles were reported in PCT-2-77/R1-79, "Report on Phase 1A", dated January 31, 1979). Several unexplained features of the earliest thermal cycles may be reproduced and are now understood.

In the earlier heat engine an a.c. coupled infra-red detector was used to measure the temperature of the ferroelectric sample. This approach required that hundreds of thermal cycles be averaged to obtain a reasonable signal-to-noise ratio. The recent measurements, however, have been made with thermocouples whose signals have been filtered. Temperature measurements are now possible on a cycle by cycle basis.

Thermocouple thermometry allowed low frequency thermal cycles to be measured. This lower frequency, in turn, allowed the employment of a Peltier* effect module to pump heat into and out of the ferroelectric sample (in place of vapor jets).

* Cooling (or heating) may be obtained at a metal-semiconductor junction by the passage of an electrical current. This phenomenon is called the Peltier effect.

The Peltier module is driven by a bipolar power supply whose output current (0 to ± 10 amp) is proportional to an input voltage. Thus, the amount of heat delivered to the sample is well controlled in amplitude and time. Periodic heat pumping waveforms (of any reasonable shape) are generated by a TRS-80 computer.

As a preliminary to our improved thermal cycle measurements, a number of electrocaloric experiments were performed. Initial measurements appeared to disagree with the temperature changes (under adiabatic conditions) predicted by Devonshire theory. Many tests were performed on the temperature measuring technique to insure its accuracy. A resolution to the apparent difficulty has been suggested which is of sound physical basis. The explanation predicts that above the Curie temperature the experimental and predicted temperature changes will be identical. This will, of course, be tested as soon as possible.

Having introduced the recent observations of the electrocaloric effect, it may be stated that the thermal efficiency (ignoring hysteresis and resistive losses) of the cycles we have produced is roughly:

$$\Delta T \text{ (electrocaloric)} / 2T = .05\%$$

(for our largest measured electrocaloric charge of 0.3°C). We will certainly achieve an efficiency of .2% ($\Delta T = 1.2^\circ\text{C}$ was reported by Thatcher¹) and are hopeful of even greater efficiency.

As a final note, investigations into low loss hysteresis loops have begun in earnest with the construction of a 100,000 Henry inductor (for inductive reduction of hysteresis as proposed by Dr. Drummond). This enormous inductor is necessary when experimenting with small samples of ferroelectrics at low frequencies. Also, we are about to begin testing a number of heat switching approaches which will be vital to the operation of a two stage heat engine.

The detailed discussions which follow begin with an explanation of the new experimental approach. Later, in section III a discussion of results will be presented. Finally, the conclusions and future plans will be presented in the fourth and fifth sections.

II. EXPERIMENTAL APPROACH

2.1 Overview

The most important experimental change which has been made in the past few months is that the signal to noise ratio of the temperature measurements has been increased to roughly ten. Previously, the signal to noise ratio on a single cycle basis was less than unity. Key to the improved temperature sensitivity was the incorporation of low frequency (sub-Hertz) thermocouple thermometry in place of high frequency (>10 Hz) unchopped infra-red detection.

After a brief look at the broad organization of the experiment the individual units will be discussed separately. An overview of the thermal cycle experiment is presented by the block diagram of Figure 1. The heart of the experiment is the sample shown in the middle of the figure. The sample receives controlling inputs from the "drivers" which appear in the upper half of the diagram. The output responses of the sample are sensed, amplified and recorded by the "blocks" in the lower portion of the figure. The individual boxes of Figure 1 will now be detailed.

Fig. 1 BLOCK DIAGRAM OF EXPERIMENT

2.2 Signal Generation

Two forms of signal generators have been built and used. The first system to work was an analog type. Two signals of different frequency, one for high voltage control another for thermal control, were required. The thermal signal frequency must be twice the frequency of the high voltage signal (see original proposal PCT-2-77 dated July 29, 1977 for details). For repeatability the phase difference between the two signals must be constant (though selectable) and therefore two independent standard signal generators are not satisfactory. Instead, one standard signal generator plus a home-built frequency doubler provided the analog control of the high voltage and Peltier current. Figures 2 and 3 illustrate the analog generator. The major difficulty in using this generator was that of phase selection. Since phase was shifted using a bandpass filter, no simple "phase knob" existed. Also, not all phase differences were attainable and waveform asymmetry was a problem. In spite of these difficulties some data, as described later, were obtained using the analog generator.

The second generator, the TRS-80 plus home-built interface, Figure 4, became operational shortly after the first thermal cycles had been taken with the analog generator (thus removing the need to improve the analog generator). The computer system generates two generalized periodic waveforms of selectable phase difference. Appendix A illustrates the computer usage and contains schematics of the interface unit.

Fig. 2 Schematic of Analogue Signal Generator

Fig. 3 EXPERIMENTAL BENCH

SHOWING UPPER LEFT: x-y-y PLOTTER; UPPER CENTER: SAMPLE CHAMBER AND THERMOCOUPLE AMPLIFIERS; UPPER RIGHT: HIGH VOLTAGE (± 300 V) AND HIGH CURRENT DRIVERS; LOWER LEFT: FREQUENCY DOUBLER; LOWER RIGHT: HIGH VOLTAGE POWER SUPPLY (± 1000 V DRIVER IS KEPT OUTSIDE OF SCREEN ROOM).

Fig. 4(a) TRS-80 COMPUTER AND P.C.T. BUILT INTERFACE

Fig 4(b) DETAIL OF INTERFACE UNIT

2.3 High Voltage Driver

High voltage is the first two "inputs" which are required by a ferroelectric sample in order to obtain a heat engine thermal cycle. Initially, attempts were made to acquire a commercial Bipolar high voltage power supply which was remotely programmable by voltage*. PCT was unable to obtain (by leasing, buying, or borrowing) such a device.

As a result, two high voltage drivers were built in-house. Figures 5 and 6 are schematics for these two drivers. The ± 300 volt transistor circuit of Figure 5 is, of course, limited in its applicability due to its low voltage swing. Most 0.25 mm thick ferroelectric samples require 500 to 1000 volts to saturate their polarizations. The relay controlled driver circuit of Figure 6 is capable of the high voltages (up to ± 1200 volts). This driver was used for measurements included in this report (with the single exception being the frequency response experiment of Figure 12).

The relay controlled high voltage driver generates exponential voltage waveforms (by charging an RC, first plus then minus). One result of this arrangement is that the voltage amplitude is a function of frequency. Another result which is livable but not desirable is that the voltage waveform is influenced by the ferroelectric sample behavior**.

* KEPCO (Flushing, N.Y.) builds such a device occasionally. PCT has been promised delivery of two such units by February, 1980 after KEPCO's next production run.

** We are presently "boxing" yet a third high voltage driver, ± 750 V which will serve until our KEPCO units arrive.

Fig. 5 ± 300 Volt Driver

Fig. 6 ALTERNATE EXPONENTIAL HIGH VOLTAGE DRIVER ($\pm 1200\text{ V}$)

2.4 Peltier Effect Thermal Drive

The second "input" which a ferroelectric sample requires for a thermal cycle is heat. The injection and removal of heat during the cycle must be well controlled with respect to timing and amplitude. In terms of control, a Peltier thermoelectric module (as illustrated in Figure 7), serves the heating and cooling function very well. The Peltier modules (Cambion Corporation # 3958-01) which are used may transfer as many as four watt/cm² and can operate at temperatures up to 124°C. The Peltier module is driven by a bipolar power supply, Figure 8, whose output current (0 to $\pm$ 10 Amp) is proportional to input voltage. The module and sample are housed in a black aluminum oven whose temperature is elevated by a heat lamp. Figure 9 shows this arrangement.

Fig. 7 EXPERIMENTAL TEST STAND

Fig. 8 HIGH CURRENT (± 10 Amp) PELTIER DRIVER

Fig. 9 SAMPLE, PELTIER MODULE AND OVEN; PARTIALLY DISASSEMBLED

2.5 Thermocouple Thermometry

Temperature measurements were made using chromel-constantan thermocouples. This type of thermocouple generates roughly sixty microvolts per degree centigrade temperature difference. With broad band (10 KHz) amplification the electrical noise is equivalent to approximately two-tenths of one degree centigrade. This noise amplitude should be compared with signal amplitude which is typically measured. The temperature swings which have been encountered in the electrocaloric measurements have been roughly one-tenth of one degree centigrade. Thus, the signal to noise ratio is sub-unity for the broad band thermocouple measurement (which is comparable to the signal to noise for the I.R. detection scheme used for earlier experiments). This problem may be solved by limiting the bandwidth of the amplifying circuitry. Figure 10 shows a low pass filter amplifier (with a bandwidth of 20 Hz) which was used to condition the thermocouple voltage. With this circuit the electrical noise is reduced to the equivalent of 0.02°C . Further improvements in thermal sensitivity were achieved by placing three thermocouples in series. Such a triple thermocouple (thermopile) generates a three-fold larger signal with no noticeable increase in noise amplitude.

As a final point regarding noise, the electrical environment of the experiment should be mentioned. Within

Fig. 10 THERMOCOUPLE AMPLIFIERS

a thirty meter radius of the solid state heat engine experimental station are a number of high voltage experiments (including a Megavolt ion accelerator). In order to avoid the electromagnetic signals of these other activities it was necessary to construct a screen room (most of which was borrowed from U. C. Irvine) to house the heat engine experiment. The screen room, shown in Figure 11, has shown to be almost totally effective in "protecting" the most sensitive electronic circuits (the computer and the thermocouples amplifier) from outside signals.

Having solved the problems of signal to noise, the calibration was checked. The measured calibration of $350 \text{ mV}/^{\circ}\text{C}$ for the thermocouple-amplifier combination agrees with that calculated by the product of the standard thermocouple calibration and the amplifier gain.

The final point on thermometry concerns that of thermal flow. Was the thermocouple actually measuring the temperature of the sample? Good temperature measurements require that the thermocouple be at equilibrium with the sample. Figure 12 shows a series of electrocaloric measurements (described in Results) as a function of frequency. Clearly, measurements at 1 Hz and above should be avoided for the thermocouple arrangement used here. A number of different methods were used to attach thermocouples to the ferroelectric samples (including epoxy, epoxy using a mica pad

Fig. 11 SCREEN ROOM

Fig. 12 / ELECTROCALORIC MEASUREMENT AS A FUNCTION OF FREQUENCY
(EMCÓM U-60)

for electrical insulation, vacuum grease, 4 mil scotch tape and 2 mil Kapton tape) and all measurements gave identical temperature swings as long as the frequency was less than one-half Hertz. This is reasonable since the characteristic penetration length for a temperature profile in a very poor thermal conductor is about one millimeter (for a time period of one second), i.e. four times as great as the sample thickness. One may be concerned that a major fraction of the heat generated in some thermal process might leave the sample before the thermocouple reaches equilibrium. Experimentally, however, the leak out processes, which are dominated by convection and radiation, have a time constant of about one minute. Thus, measurements taken in a period of a few seconds suffer only an unimportant fractional decrease.

2.6 Data Recording

Finally, the data recording boxes of Figure 1 may be described. The x-y-y recorder (Hewlett-Packard 7046A) receives the voltage of the charge measuring capacitor in its x axis. The sample voltage enters the recorder's y-1 axis and the temperature voltage from the thermocouple amplifier enters the y-2 axis. Thus, the x-y-y plotter generates an hysteresis figure and a "T" versus "Charge" figure simultaneously.

Additional information is recorded by a storage scope (Tecktronix 5113). Sample voltage, charge and temperature, as well as Peltier Module input, are stored by the scope as a function of time.

III. RESULTS AND DISCUSSION

3.1 Electrocaloric

As a preliminary to our improved thermal cycle measurements, a number of electrocaloric* experiments were performed. In an electrocaloric test one measures the adiabatic change in sample temperature as sample charge varies. The entropy density equation (3.6 on proposal page 23),

$$ds = -\gamma_0 d\sigma + \frac{C_p}{T} dt \quad (1)$$

may be used to predict the adiabatic temperature change in

That is,

$$T = \frac{T\gamma}{2C_p} \left[\sigma_f^2 - \sigma_i^2 \right] \quad (2)$$

Under these conditions the electrocaloric behavior should be as depicted in Figure 13. Electrocaloric diagrams for measurements which contain irreversible processes are given in Figures 14 and 16. Two observations are immediate. First, the scale of the temperature change in the (irreversible) measurements is lower than that "expected" by the (reversible) entropy density equation. The word "expected" is given in quotation marks since an equation derived

* These experiments should be referred to as polarocaloric measurements since the adiabatic temperature change is referenced to the polarization change, not the electric field change.

"ELECTROCALORIC" EFFECT

Fig. 13 ELECTROCALORIC DIAGRAM (REVERSIBLE PROCESSES ONLY)

Fig. 14 ELECTROCALORIC MEASUREMENT OF BaTiO_3 AT ROOM TEMPERATURE (EMCON U-60, .25 mm thick).

Fig. 15 HYSTERESIS LOOP (UNCOMPENSATED) OF BaTiO_3 (RECORDED SIMULTANEOUSLY WITH ELECTROCALORIC MEASUREMENT OF FIG. 11).

Fig. 16 SIMULTANEOUS PLOT OF ELECTROCALORIC MEASUREMENT AND HYSTERESIS (UNCOMPENSATED) LOOP AT ROOM TEMPERATURE. SAMPLE IS PZT (Vernitron PZT-5H).

for reversible processes cannot be relied upon to predict temperature changes for an irreversible process. None the less, it is useful to compare magnitudes.

Using equation (2) for the PZT case, $\Delta\sigma = 0.3 \text{ C/m}^2$, $\gamma = .3 \text{ Megavolt-meter/C}^\circ\text{K}$, $C_p = 3 \times 10^6 \text{ J/m}^3\text{K}$, and $T = 300^\circ\text{K}$, yielding a temperature change, ΔT , of 1.5°K . The measured value of ΔT much below the Curie point, where irreversible processes are dominant, is less than one-tenth as large.

The discussion of pages 25 through 27 of the January 21, 1979 report points out that the difference between the reversible and irreversible case is a matter of charge density accounting. One may apply an equation identical to equation (1) even in an irreversible case, but there one must sum up entropies for many regions. The result is that when a measured charge density, equal to $\Sigma\sigma_i$, is squared -- giving $(\Sigma\sigma_i)^2$ -- the result is not necessarily equal to the sum of the squares of the individual charge densities, $\Sigma(\sigma_i^2)$, (which is the sum by which entropy may be calculated). Physically, as a ferroelectric is forced around a hysteresis loop, the dominant charge changing event is that of polarization flipping. Domain by domain, σ_i changes to $-\sigma_i$, and therefore σ_i^2 remains constant, thus giving no change in entropy. The small amount of charge change which occurs outside of the hysteresis loop does involve a change in σ_i and therefore contributes a small entropy change (which is reflected as a small temperature change in an adiabatic

measurement).

The second observation of note is that the electrocaloric diagrams are not always parabolas. Non-quadratic behavior is particularly evident in the Barium Titanate diagram of Figure 14. As yet the authors are unaware of any convincing explanation as to the origin of the loops in the diagram, but should mention that they bear some resemblance to Strain versus Polarization diagrams for ceramic barium titanate².

3.2 Thermal Cycle

Setting aside, for the moment, the difficulties in the low temperature electrocaloric measurements, one may address a demonstration of a solid state heat engine thermal cycle. What happens to the temperature behavior shown in Figure 13 if heat is added and extracted from the ferroelectric sample in a Carnot fashion? The result, shown in Figure 17, may be described as follows. Starting at the point 0, heat is added to the sample as the ferroelectric begins to discharge. When the charge is reduced to 70% of its initial value, at point 1, heating is stopped while discharging continues. At the end of the adiabatic discharge, point 2, an isothermal charging begins (with external cooling required). At point 3 the sample is again thermally isolated while charging continues. Point 4 is essentially the same as point 0, but with the polarization pointing in the opposite sense. The next cycle then proceeds via points 5, 6, and 7 to return to 0. The journey just described corresponds to twice around an ideal carnot rectangle of T vs. S space. Note that any inequivalence of the two wings of this thermal cycle (not to be confused with the butterfly loops of barium titanate) will result in a double looped T vs. S diagram.

Figure 18 shows a thermal cycle, obtained by Peltier effect heating and cooling. Asymmetries in the hysteresis loop, along with asymmetries in the heating and

HEAT ENGINE THERMAL CYCLE

Fig. 17 HEAT ENGINE CARNOT CYCLE

Fig. 18 THERMAL CYCLE
SAME SAMPLE AS THAT OF Fig. 16
(ANALOG SIGNAL GENERATOR).

cooling function, result in the unbalanced pair of loops.

Shown in Figure 19 is a thermal cycle obtained using a sample of PZST. Also illustrated in Figure 19 is an electrocaloric measurement which was taken with the sample mounted to the PELTIER module (rather than isolated as for a genuine electrocaloric measurement).

It should be emphasized that a series of cycles, similar to that illustrated in Figure 18, were taken on two separate days and repeated very nicely.

Fig. 19 SEMIELECTROCALORIC (LOWER TRACE) AND
THERMAL CYCLE (UPPER TRACE) OF PZST - B.T. 50/45/5
SAMPLE TEMPERATURE 48.6°C, OBTAINED WITH COMPUTER CONTROL

IV. CONCLUSIONS

In summary, it has been seen that the electrocaloric behavior measured below the Curie temperature differs greatly from that calculated by reversible thermodynamic considerations.

It is expected that when measurements are taken above the Curie temperature -- where hysteresis becomes small and thus reversibility applies -- that the electrocaloric behavior will be predictable. Such a change has been noted previously and is shown in Figures 20 and 21, taken from Wiseman³.

The largest electrocaloric swing which has been measured in this investigation was roughly 0.3° for a sample which underwent a ferroelectric to anti-ferroelectric transition (PZST B.T. 50/45/5). This corresponds to an efficiency (neglecting hysteresis losses) of

$$\Delta T \text{ (electrocaloric)} / 2T = .05\%$$

The more complete work by Thacher on samples of this type has shown $\Delta T = 1.2^\circ\text{C}$.

Fig. 20. Dielectric and electrocaloric measurements of KH_2PO_4 above the Curie temperature. Values of ΔT are cumulative, and the resulting error of closure is a measure of cumulative errors and a net loss of heat.

Fig. 21. Dielectric and electrocaloric measurements below the Curie temperature. The electrocaloric effect in KH_2PO_4 below the Curie temperature is a linear function of the applied field beyond the low-field region where irreversible (hysteresis) effects are large.

V. FUTURE PLANS

First on our list of future activities is, of course, to investigate the electrocaloric effect at temperatures above the Curie point in order to verify the prediction of reversible thermodynamics. In the process, larger electrocaloric changes will be found. The actual measurements in this effort will be performed at both P.C.T. and the University of Illinois.

Secondly, loss mechanisms -- and how to avoid them -- must be dealt with. At present, hysteresis loss swamps power production. Serious experiments on inductive hysteresis reduction will soon begin. Using a non-Carnot cycle it may be possible to avoid hysteresis completely and increase the working temperature range.

Finally, the primary objective during the next year will be the development of thermal switches which will serve in a two-stage heat engine. This may prove to be a formidable task limiting our ability to push on the activities mentioned earlier. However, early tests indicate that the thermal switching problem has been essentially solved for sub-Hertz operation. Our outlook for solving the deeper problems of a Solid-State Heat Engine is therefore very optimistic.

APPENDIX

This appendix contains a pair of examples illustrating the computer system waveform generator and a set of schematics which detail the output interface.

Included in the first example is the text of a "conversation" between the computer and the experimenter. The experimenter's inputs have been underlined. The first two figures are photographs of the computer T.V. display. Function 1 (Fig. A1) is a sine wave generated by the computer. Function 2 (Fig. A2) is an arbitrary function which has been entered point by point from the keyboard following the computer's instructions on functions drawing (see text). Fig. A3 then shows the interface output of the two functions as they appear on an oscilloscope. The second example shows a pair of truncated sine waves as they appear on the T.V. (Figs. A4 and A5) monitor and oscilloscope (Fig. A6).

In the computer synthesized mode (any truncated sine wave), the second function is automatically generated at twice the frequency of the first function as required for thermal cycles (see section 2.1).

EXAMPLE 1

TRSDOS - DISK OPERATING SYSTEM - VER 2.2

CLOCK

DOS READY

LOAD FUNCTGEN/CMD

DOS READY

HOW MANY FILES? (RETURN)

MEMORY SIZE? 40000 (RETURN)

RADIO SHACK DISK BASIC VERSION 2.2

READY

LOAD "FUNCTGEN/TXT" (RETURN)

READY

RUN (RETURN)

FUNDAMENTAL FREQUENCY OF BOTH WAVES IN Hz? 0.2 (RETURN)

USE ↑ & ↓ WITH THE SPACE BAR TO DRAW ONE CYCLE

(+ DELETES, / STORES; 1 AND 2 DISPLAY STORED CYCLE

1 OR 2). ALTERNATIVELY A TRUNCATED SINEWAVE CAN BE USED.

PRESS G WHEN YOU ARE READY TO GO ON THE DRAWING.

IF YOU WANT THE SINEWAVE, PRESS S. S

AT WHAT PERCENTAGE OF AMPLITUDE DO YOU WANT SINE

TRUNCATED? 100 (RETURN)

WHAT PHASE DISPLACEMENT (IN DEGREES) DO YOU WANT? 90. (RETURN)

Fig. A1 FUNCTION #1 (Port #1). FUNDAMENTAL FREQUENCY = .2 Hz

Fig. A2

PRESS G WHEN YOU ARE READY TO GO ON DRAWING FN2.
IF YOU WANT THAT TO BE A TRUNCATED SINEWAVE, PRESS S.

FUNCTION #2 (Port #0). FUNDAMENTAL FREQUENCY = .2Hz

BREAK IN 21090.

READY

Go to 500.

Fig. A3 OUTPUT

As seen on oscilloscope (Voltage amplitude is 10 Volt+)

EXAMPLE 2

TRUNCATED SIN (90% of SINWAVE)

Fig. A4 FUNCTION #1 (PORT #1). FUNDAMENTAL FREQUENCY = 10 Hz.

TRUNCATED SIN (50% of SINWAVE)

Fig. A5 FUNCTION #2 (PORT #0). FUNDAMENTAL FREQUENCY = 10 Hz

Fig. A6

Interface output as displayed by oscilloscope

The computer interface components which are required to convert the TRS-80 digital output into a usable laboratory voltage are illustrated in the schematics of Figures A7 through A9.

To Computer

To Interface

N74368 Driver Chips

Fig. A7 INPUT-OUTPUT BUFFER DRIVERS

Fig. A8 ADDRESS DECODER

Fig. A9 DIGITAL TO ANALOG CONVERTER

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