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MEASUREMENTS OF PYROELECTRIC RESPONSE OF TGS SINGLE CRYSTALS USING THE COMPENSATION METHOD

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Abstract: An alternative method for the precise measurement of temperature characteristics of pyroelectric response in TGS single crystals is presented. The paper contains a brief description of the measurement system using a digitally controlled compensation method. Sample measurements are presented and discussed.

Keywords: pyroelectric effect, measurement, compensation method.

1. INTRODUCTION

Microphones, quartz watches, inkjet printers, all rely on a phenomenon known as the piezoelectric effect. This phenomenon can be found in various materials both organic and non-organic. Piezoelectricity was first discovered by Pierre Curie, who found that putting pressure on quartz crystal created electricity. He put his discovery to good use by inventing the piezoelectric charge meter, built around 1889, it was used in the discovery of radium and polonium [1]. It was also used in one of the first instrumental research of pyroelectricity. The idea of measurements of small charges, using the compensation method, is recommended especially in the case of precise measurements and relatively slow processes. We decided to "digitalize" the idea of the Curie method in our investigations of pyroelectric response of TGS single crystals. The idea of the Curie method is relatively simple and presented in figure 1. As one can see, the charge produced by the pyroelectric crystal is balanced by the charge generated by force applied to the model piezoelectric single crystal. The state of equilibrium is traced by electrometer. The force applied to the model sample is proportional to the electric response of the examined sample.

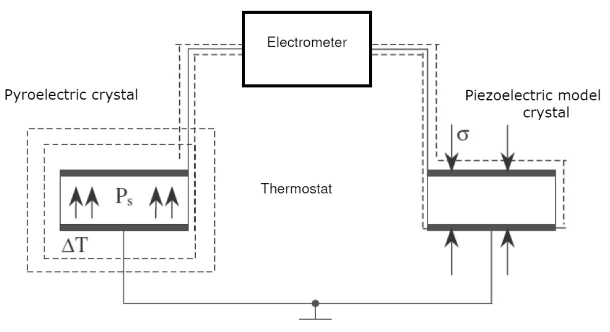

Fig. 1. Historically the first method to measure the pyroelectric coefficient of the Rochelle salt sample used by the Curie brothers

TGS (Triglycine Sulphate) is an organic ferroelectric material that can be obtained by growing it from water solution. Its properties and mechanisms of the phase transition have been investigated by numerous authors since the moment of discovery of its peculiar properties [2-3]. Investigation of pyroelectric response and the mechanism of the phase transition is important from the point of view of its application in infrared active sensors. There are numerous methods of investigation of pyroelectric response of TGS samples [4]. We decided to use the compensation method, having its origin in the Curie brothers piezoelectric electrometer idea. In our system, the signal proportional to the pyroelectric charge, produced by the sample, is compared with voltage measured on a standard capacitor that can be charged or discharged by digitally controlled current sources.

2. OPERATION OF THE MEASURING SYSTEM

The system records signals proportional to the samples' charge response caused by the change of temperature. All calculations (spontaneous polarization, pyroelectric current) are made in post processing. Data transmission between computer and measuring device is realized using an RS 232 computer port and created software. The system consists of the functional blocks presented in figure 2.

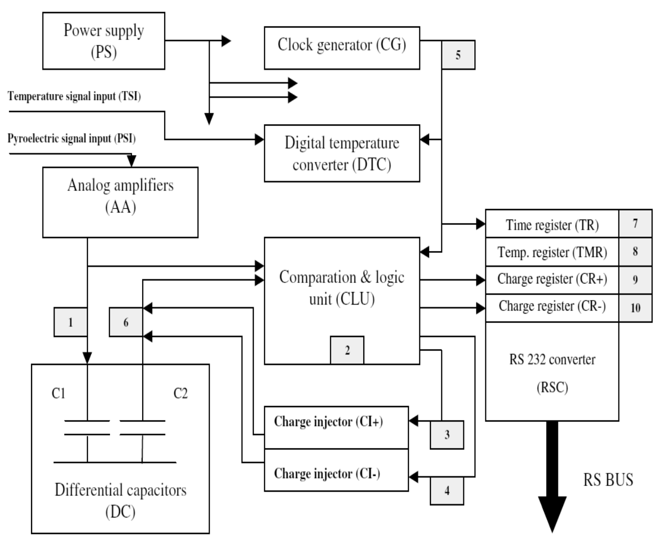

Fig. 2. Block diagram of the measuring system

The role and markings of functional blocks presented in figure 2 are as follows:

Power Supply (PS) – supplies all voltages necessary for powering the system, Pyroelectric Signal Input (PSI) – high resistance input of the pyroelectric charge signal, connected to the ferroelectric TGS sample mounted in a thermostatic unit, Temperature Signal Input (TSI) – voltage input connected to a temperature sensor, Analog Amplifiers (AA) – signal conditioners for charge input, Differential Capacitors (DC) – a set of two capacitors, voltage U_1 on C_1 is proportional to the pyroelectric charge on the sample, voltage U_2 on C_2 is changed by charge injectors, depending on the balance between U_1 and U_2 (the processes of voltage comparison and logical signal generation that control the charge injectors are carried out by the Comparison and Logics Unit – CLU), Clock Generator (CG) – generates time impulses needed to synchronize the system. The CG signal parameters are presented in Fig. 3. Digital Temperature Converter (DTC) – makes analog to digital conversion of temperature signal, Comparison and Logic Unit (CLU) – compares U_1 and U_2 voltages and, depending on the results, generates impulses that control the charge injectors, Charge Injectors (CI_+ and CI_-) – digitally controlled sources of impulse current, that inject a charge to C_2 in order to preserve the relation of $U_1 = U_2$, Time Register (TR) – counts time impulses, Temperature Register (TMR) – records temperature values, Positive Charge Register (CR_+) – stores the accumulated value of positive charge pulses; Negative Charge Register (CR_-) – stores the accumulated value of negative charge pulses; RS Converter (RSC) – converts the values of the registers and controls the transmission of measurement data via the RS 232 port.

3. DATA PROCESSING

The ferroelectric sample is placed in a thermostat and connected to the high resistance input of AA. The signal from the sample is amplified *(K=100–1000)* and transmitted to the C_1 capacitor. Voltages U_1 on C_1 and U_2 on C_2 are compared. If the voltage U_1 is not equal to U_2, the CLU generates proper impulses for the CI units and C_2 is charged or discharged until the relation $U_1 = U_2$ is regained. Impulses are counted by the CR units. Value of the charge injected to C_2 depends on the CG signal width *(17.7 ms)* and adjustable amplitude of the current impulse *(0.2–0.8μA)*. the system stores values from all the registers (TR, TMR, CR_+, CR_-).

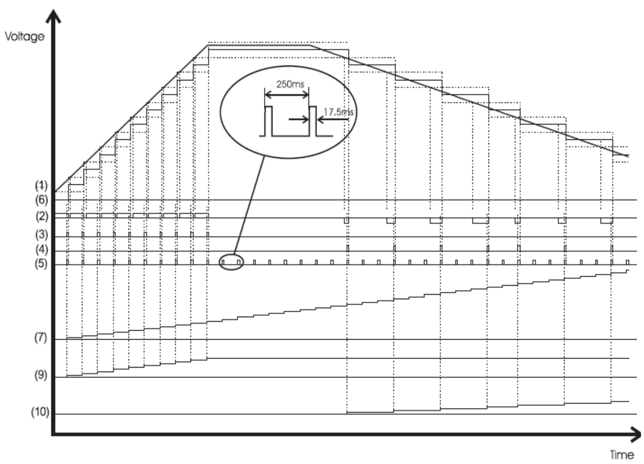

Fig. 3. Time waveforms of selected analog and digital signals, illustrating the principle of operation of the system. Simulation made for trapezoidal voltage input. Waveform numbers correspond to node numbers presented in figure 2

In this manner, it is possible to trace changes in the samples' polarization versus time and temperature. With use of the registered data, one can calculate the temperature characteristics of pyroelectric current and pyroelectric coefficient in post processing.

Time waveforms of selected analog and digital signals generated by the system during data acquisition are presented in figure 3.

4. SYSTEM CALIBRATION AND MEASUREMENTS

Standard - aged sample of TGS was heated to 60˚C and left in thermostat for 30 minutes. The rejuvenated sample, after cooling down to room temperature, was used in measurements of temperature characteristics of pyroelectric current with use of traditional measurement (recording of the electric current in short circuit of the sample). The sample was heated and cooled three times and pyroelectric current together with temperature were recorded.

The treated sample was left at room temperature for aging. Measurement of the polarization using the compensation method was made after 24 hours. Before the measurement, the sample was rejuvenated again.

Temperature and charge signals were connected to TSI and PSI terminals respectively. Signals of digital counters CR_+ and CR_- proportional to change of the charge on sample plates were recorded along with the time and temperature signal. Values of the charge counters were used to obtain time and temperature dependencies of polarization, using the equation:

$$P(T) = \frac{CR_+ - CR_-}{S} \cdot \Delta q \quad (1)$$

where, *P(T)* - actual value of polarization, $CR_{+/-}$ - actual values of charge registers Δq - constant value depending on selected resolution of charge injectors - calculated as length of current impulse *(17,5 ms)* multiplied by current amplitude *(0,2–0,8 μA)*, *S* – surface of the sample.

LabVIEW software was created in order to collect and observe measurement data on-line in various aspects. Screen shots of the system operation are presented in figures 4 and 5. In figure 4, one can observe the differential plot of the charge registers (CR_+ and CR_-) during 3 heating and cooling processes, according to the temperature excitation diagram presented in figure 5.

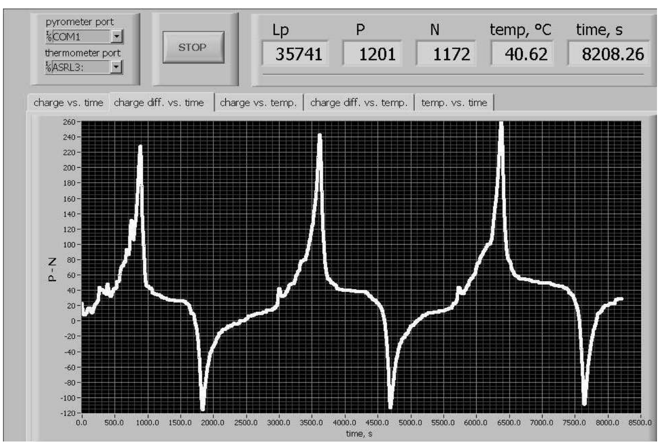

Fig. 4. Three cycles of heating and cooling registered with using the digital compensation method

Calculation of pyroelectric current based on registered polarization signals was performed and the values were compared with those obtained using a traditional method. In this way, we were able to calibrate our digital compensation electrometer.

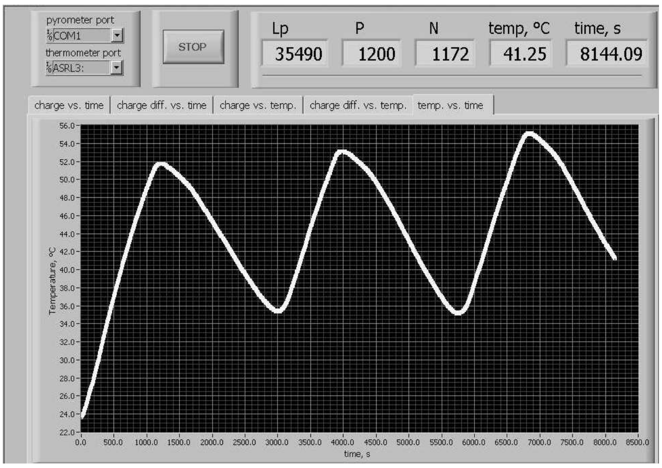

Fig. 5. Temperature profile applied to the sample after rejuvenating

Measurement results after the calibration process are presented in figure 6 where one can observe plots of pyroelectric current obtained using two methods. The presented results are one of many samples included in the measurements.

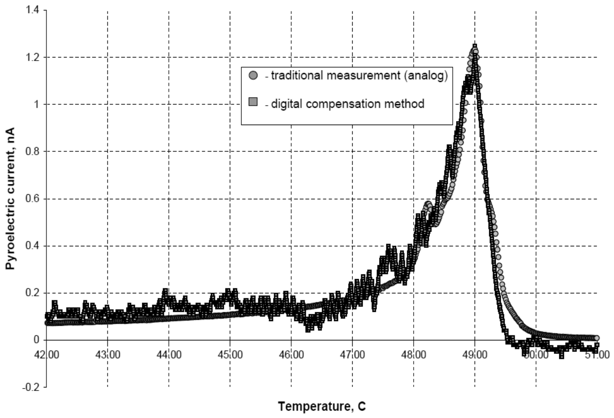

Fig. 6. Results of measurement of pyroelectric current, close to the phase transition temperature Tc=49°C, obtained with using two methods after calibration of newly created system

5. CONCLUSIONS

Firstly, the measurement system was designed and constructed. In order to calibrate the device, we investigated the pyroelectric phenomenon using both the traditional and new method simultaneously. Because of a significant nonlinearity of pyroelectric phenomenon in TGS samples, the new system was calibrated in the vicinity of the phase transition temperature. A KEITHLEY 485 Electrometer was used in the calibration process.

The presented method assures higher sensitivity when compared with traditional methods. It enables the observation of ferroelectric domain switching near the point of phase transition vs. time and temperature. Multiple pyroelectric charge peaks could be observed with using the compensation method (figure 6). Such observations were nearly impossible using the traditional method of pyroelectric current measurement [5]. This can be attributed to the discharge of the sample due to the current flowing through the resistance of the picoammeter used in traditional method, which is about three orders of magnitude smaller than the resistance of the TGS sample. Input resistance of our device is comparable with the resistance of the sample. We hope the new measurement method can be utilized in research into the design and construction of better ferroelectric infrared sensors.

6. REFERENCES

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POMIARY ODPOWIEDZI PIROELEKTRYCZNEJ MONOKRYSZTAŁÓW TGS Z WYKORZYSTANIEM METODY KOMPENSACYJNEJ

W artykule przedstawiono alternatywną metodę pomiarów temperaturowych charakterystyk odpowiedzi piroelektrycznej monokryształów TGS. Metoda pomiarowa oparta jest o ideę elektrometru kompensacyjnego zaprojektowanego i wykonanego w roku 1889 przez braci Curie. Metoda kompensacyjna jest szczególnie przydatna w przypadku precyzyjnych pomiarów sygnałów wolnozmiennych. Publikacja zawiera krótki opis zasady działania układu pomiarowego, jego schemat blokowy oraz przykładowe przebiegi wybranych sygnałów generowanych przez układ pomiarowy. Opisano procedurę kalibracji układu z wykorzystaniem elektrometru KEITHLEY 485. Wykonane zostały pomiary odpowiedzi piroelektrycznej próbek TGS, z wykorzystaniem tradycyjnej metody pomiaru prądu w obwodzie zwartej próbki, oraz z wykorzystaniem metody kompensacyjnej. Przykładowe wyniki pomiarów zostały zaprezentowane. Przeprowadzono porównanie otrzymanych wyników.

Keywords: efekt piroelektryczny, pomiar, metoda kompensacyjna.