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pyroelectric and dielectric properties of single triglycine sulphate crystal under different poling temperatures

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الخواص الكهروحرارية والعزلية لبلورة كبريتات ثلاثي الجليسين المفردة عند درجات حرارة استقطاب مختلفة

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Abstract
In the present work, dielectric constant measurements in the vicinity of the phase transition point (Tc) ≈49ºC and so pyroelectric current measurements after poling process at different temperatures and constant poling field of 4kv/cm subjected on samples of triglycine sulphate were cut from a large single crystal. The results show that the maximum value of the dielectric constant at the transition temperature (Tc) decreases with increasing the frequency and that the peak shifts to lower temperatures and it was found that by increasing the poling temperature the peak of pyroelectric current increases and shifts to lower temperatures.

Keywords: pyroelectric, triglycine sulphate, poling process.

الملخص
في هذا العمل، أُجريت قياسات ثابت العزل الكهربائي بالقرب من نقطة انتقال الطور (49 درجة مئوية) ، بالإضافة إلى قياسات التيار الكهربائي الحراري بعد عملية استقطاب عند درجات حرارة مختلفة، ومجال استقطاب ثابت قدره 4 كيلو فولت/سم، على عينات من كبريتات ثلاثي الجليسين قطعت من بلورة مفردة كبيرة. أظهرت النتائج أن القيمة القصوى لثابت العزل الكهربائي عند درجة حرارة الانتقال الطوري تتناقص مع زيادة التردد، وأن ذروة التيار الكهربائي الحراري تنزاح إلى درجات حرارة منخفضة. وقد وُجد كذلك أنه بزيادة درجة حرارة الاستقطاب، تزداد ذروة التيار الكهربائي الحراري وتنزاح إلى درجات حرارة منخفضة.

الكلمات المفتاحية: كبريتات ثلاثي الجليسين، التيار الكهرو حراري، عملية الاستقطاب.

Introduction

Triglycine sulfate (TGS) is a well known ferroelectric material that is easy to grow in a form of single crystal and it is a type of order-disorder ferroelectrics with chemical formula $(NH_2CH_2COOH)_3H_2SO_4$.[1,2,3]

Triglycine Sulfate (TGS) is a versatile ferroelectric material that has been widely studied for its unique combination. It possesses several properties that make it an attractive choice as a ferroelectric material, including a high dielectric constant, a relatively low Curie temperature, and a strong pyroelectric effect. The high dielectric constant of TGS, which can be as high as 200 at room temperature, makes it a suitable candidate for applications that require a high level of sensitivity, such as infrared detectors and capacitive sensors. The low Curie temperature of TGS (around 49°C) is advantageous for applications that require a stable performance over a wide temperature range, as the material can maintain its ferroelectric properties even at relatively high temperatures. One of the most significant properties of TGS is its strong pyroelectric effect, which refers to the generation of an electric charge in response to a change in temperature. This effect is particularly pronounced in TGS, making it a popular choice for the fabrication of pyroelectric detectors, which are widely used in thermal imaging and non-contact temperature measurement applications. [4,5]

Ferroelectric TGS crystal has been extensively studied both in pure state and doped and a great deal of work has been done on its in the past two decades and the efforts are mainly focused to improve its ferroelectric, pyroelectric, piezoelectric, mechanical and optical properties .

Naveen Kohli *et.al*.[6] studied " Theoretical Study of Dielectric Properties in Triglycine Sulphate Crystal".

Ranjan Kumar1*et.al*.[7] studied" Influence of xylenol orange dye on structural,vibrational, thermal and luminescence properties of TGS crystals".

M. Trybus *et.al*.[8] studied" Pyroelectric response of single-crystal samples of triglycine sulphate in three dimensions".

Elena Balashova *et.al*.[9] studied "Croconic Acid Doped Triglycine Sulfate: Crystal Structure,UV-Vis, FTIR, Raman, Photoluminescence Spectroscopy, and Dielectric Properties".

M. Trybus *et.al*.[10] studied "Effect of triglycine sulphate (TGS) -material for active detectors of infrared radiation".

This work aims at studying Pyroelectric behavior of a triglycine sulphate single crystal after poling process .The most popular method of investigating the pyroelectric properties of TGS involves measurements of the pyroelectric charge, current or voltage for thin samples cut perpendicular to the polar **b** axis. After mechanical cleaving or cutting, the samples are polished and conductive electrodes are deposited on the surfaces . A sample fabricated in this way is a flat capacitor, making it possible to carry out pyroelectric investigations in only one direction, which is the most reactive in terms of the pyroelectric response of the sample (sensor).[11,12,13,14]

Material and methods

(1) Materials

The crystal was synthesized from chemically pure glycine acid and concentrated sulphuric acid in the molar ratio 3: 1 and grown in the ferroelectric phase.

(2) Preparation of test samples

On account of the high solubility of (TGS) in water, we used the wet thread method in cutting the samples from plane – parallel plates perpendicular to the ferroelectric b-axis. The crystal plates were etched by water on a wet piece of soft cloth stretched on a glass plate until the required dimensions were obtained (0.9×0.5×0.2) cm^3.Two thin copper wires were attached to the major opposite surfaces of the sample. The two current leads were pressing on the coated surfaces and kept in position by a trace of UHU glue which fixes the wire near its end to the upper side of the crystal. Good electrical contact between the wire and coated surfaces was insured by a minute speck of the conducting silver paste. In this way, loading or stressing of the crystal is kept at minimum.

(3) Dielectric constant

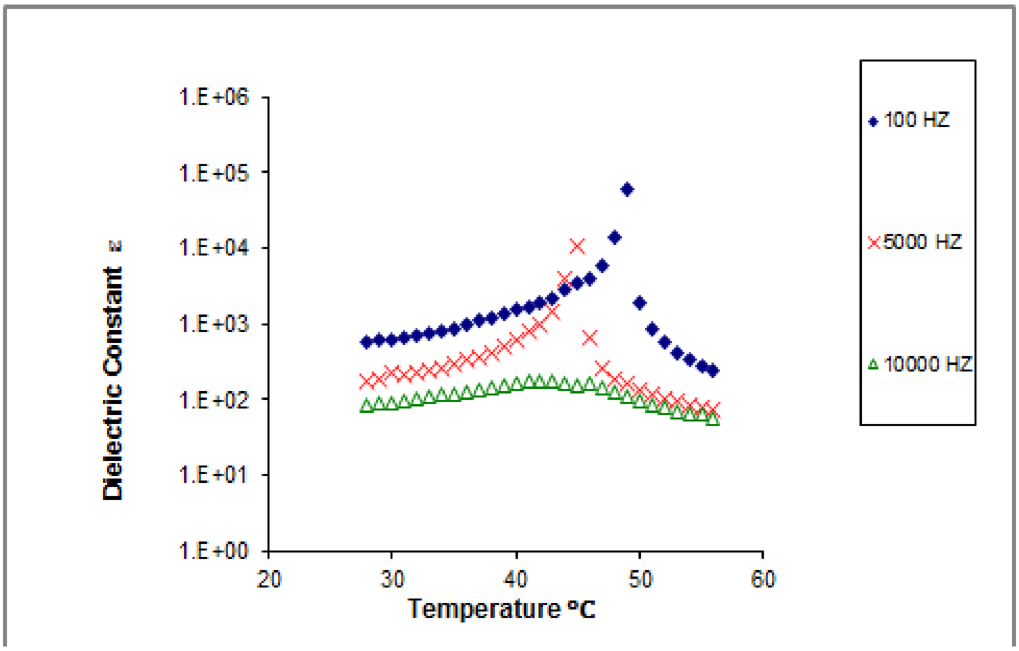
The dielectric measurements were carried out using an auto balance bridge (type GM Instek LCR-821meter) at different frequencies (100, 5000 and 10000 Hz).The measurements were carried out at temperatures ranging from 30 ºC to 60 ºC and at heating rate of (0.5 ºC /min).

(4) Pyroelectric current measurements.

The sample passed through the following process: the sample temperature was raised to [T_p: 30, 40, 50, 60, and 70 ºC], where the dipoles are able to orientate. While being subjected to constant poling voltage of about 400 volt which gives an electric field of 4kv/cm, for a time of poling of about 30 min., then the sample was cooled to room temperature in the presence of the electric field, where dipoles are assumed to be frozen. The electric field was then removed and the electrodes were shorted for removing the story surface charges induced by the poling field. In this way the sample was kept in polarized state. The pyroelectric current was recorded using an electrometer (keithley 485 auto ranging Pico ammeter), with increasing temperature by a rate of heating ≈ 2 ºC/minute.

Results and discussion

Fig. (**1**) represents the temperature dependence of the dielectric constant **ε'** for pure TGS sample in the temperature range (28 – 60 °C) at different frequencies (100, 5000 and 10000 Hz). The normal behavior was observed which gives a sharp rise in dielectric constant around a certain temperature called critical temperature (T_c) and abrupt decreases just above (T_c). We have order- disorder phase transition.



Figure(1):shows the temperature dependence of the dielectric constant (έ) for pure TGS at different frequencies.

We notice from table(1) that the maximum value of the dielectric constant $(\varepsilon^{\backslash}_{mas.})$ at the transition temperature (Tc) decreases with increasing the frequency and that the peak shifts to lower temperatures. This behavior is characteristic of dielectric relaxation governed by the finite response time of ferroelectric domain dynamics. Consequently, the polarization process becomes dynamically limited. Leading to a shift of the dielectric peak toward lower temperatures. Because the dipole may not have enough time to keep up with the changes in the field (Dielectric relaxation).[15]

Table (1) shows the maximum value of the dielectric constant $(\varepsilon_{\max.})$ and critical temperature (T_C) at different frequency (*f*)

Frequency (*f* Hz)	**Dielectric constant** $(\varepsilon^{\backslash}_{mas.})$	**critical temperature (T_c)**
100	$1.59*10^4$	49
5000	$1.10*10^4$	45
10000	$57.3*10^2$	43

Figure (2) illustrates the temperature dependence of the pyroelectric current for TGS crystal using different poling temperatures at constant poling field of 4kv/cm and The polarization process is fundamental and essential for improving and developing the thermoelectric properties of the material and The current magnitude and peak position are subject to slight changes caused by the polarization temperature. It was found that by increasing the poling temperature the peak of pyroelectric current shifts to lower temperatures. i.e. lower critical temperature Tc.

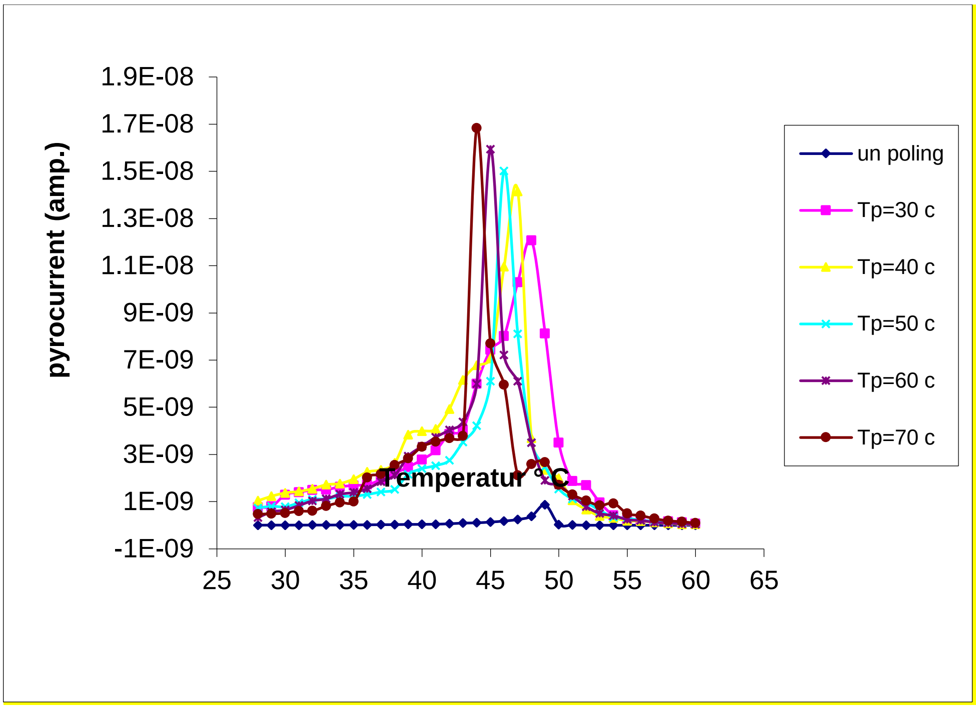


Figure (2): shows the temperature dependence of pyroelectric current for TGS sample at different poling temperatures.

Figure (3) illustrates the relationship between the maximum pyroelectric current (Imax) and the polarization temperature at Tc. The maximum pyroelectric current exhibits an upward trend corresponding to an increase in the polarization temperature. A saturation state in polarization can be achieved at high poling temperatures. Achieving a suitable polarization temperature is important for achieving improved crystalline properties suitable for various applications.

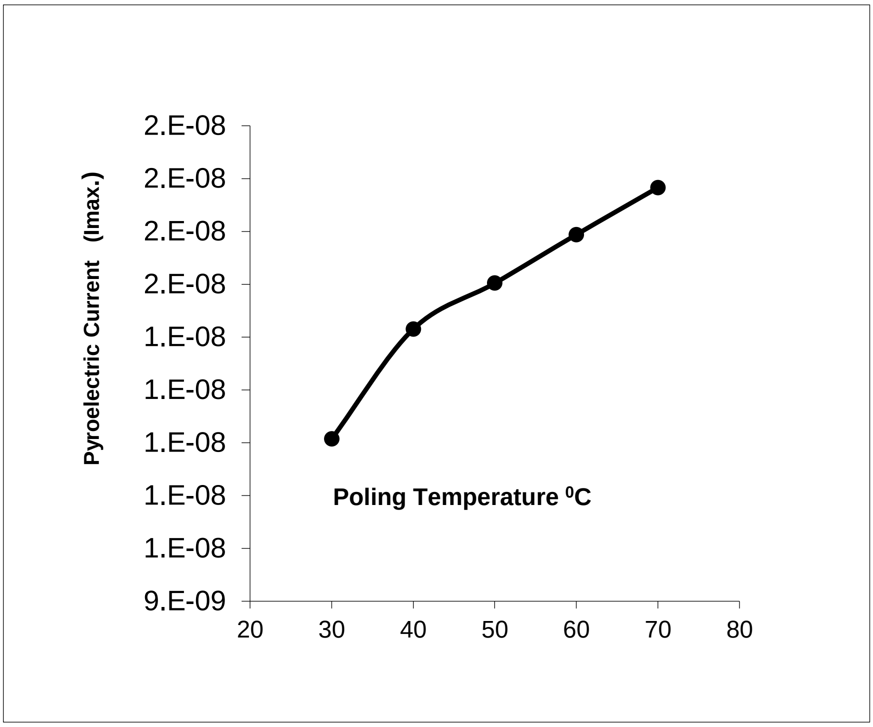


Figure (4):shows the pyroelectric current versus poling temperature at temperature below transition temperature (TC).

While below and above curie temperature there are some disturbance in the value of the pyroelectric current Figure (4),(5). The observed irregularities may be associated with residual polarization remaining in clamped domains.

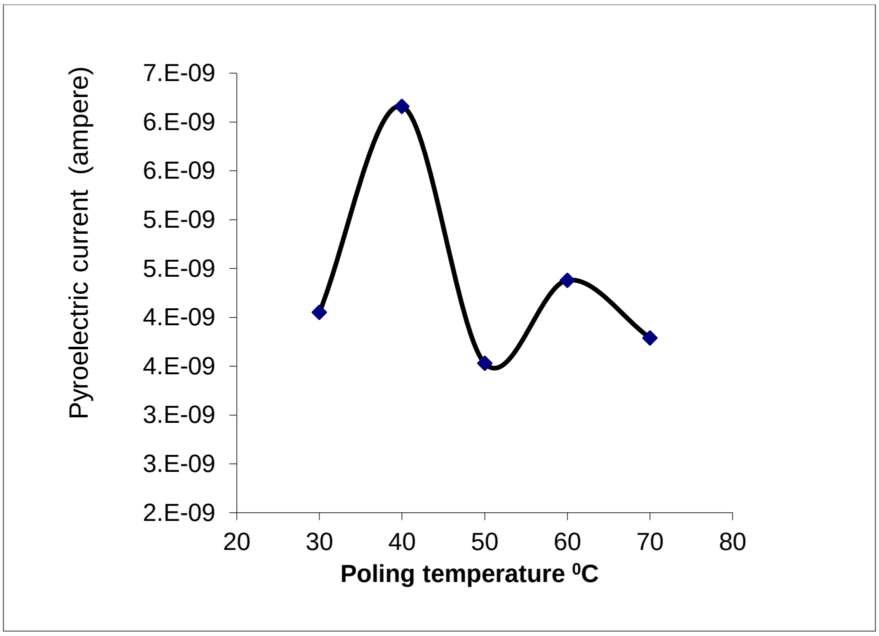


Figure (4):shows the pyroelectric current versus poling temperature at temperature below transition temperature (TC).

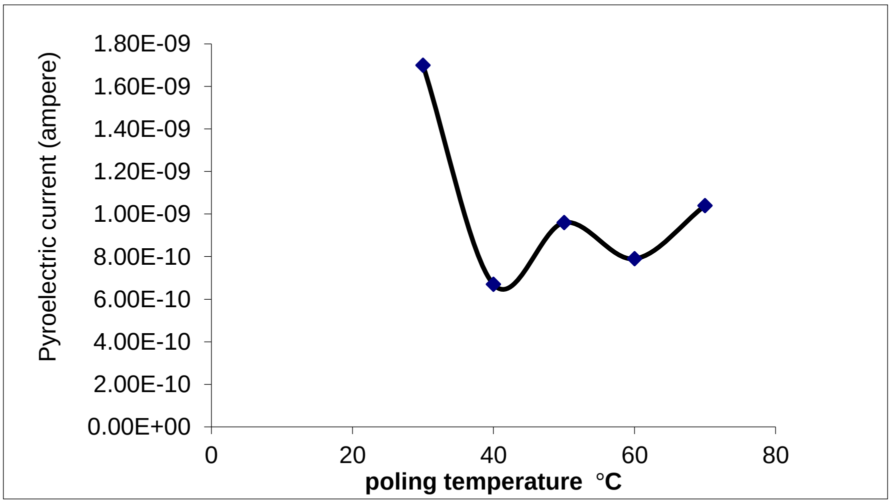


Figure (5): shows the pyroelectric current versus poling temperature at temperature above transition temperature (TC).

Table (2) shows the relation between poling temperature and transition temperature. Where decrease in transition temperature Tc is being noticed.

poling temperature (T_P ˚C)	30	40	50	60	70
Transition temperature (T_c ˚C)	48.7	47.5	46.7	45.3	44.8

The decrease in transition temperature (Tc) with increasing poling temperature may be attributed to enhanced dipole alignment and domain stabilization which modifies the internal field and slightly affects the thermodynamic stability of the ferroelectric phase. It may concluded that increasing of poling temperature assist in removal of residual polarization.

Conclusion

The following conclusion may be drawn from the obtained results that:

(1) increasing the frequency lead to decreasing dielectric constant value and the peak shifts to lower temperature .This behavior is attributed to dielectric relaxation .

(2) the pyroelectric current increasing drastically with poling temperature (T_p), because Increasing of the poling temperature assist in removal of residual polarization.

Compliance with ethical standards

Disclosure of conflict of interest

The author(s) declare that they have no conflict of interest.

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