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TRIGLYCINE SULPHATE (TGS) – MATERIAL FOR ACTIVE DETECTORS OF INFRARED RADIATION

In the article Tryglicine Sulphate (TGS) is presented – one of the iconic materials used in thermal detectors of infrared radiation (IR). In this work the second order phase transition in TGS is also demonstrated. Methods of measurements of essential electric parameters of TGS based IR sensors are presented and discussed here regarding their application in thermal/pyroelectric detection of IR radiation.

Keywords: Tryglicine Sulphate, Pyroelectric sensors, TGS

1. INTRODUCTION

Electromagnetic waves with different frequency ranges and associated energy can interact with the matter in different ways. The historical division of electromagnetic waves, together with their brief characteristics and selected applications in medicine, is presented below.

Gamma-rays (Villard 1901) are generated by radioactive atoms and in radioactive decays. They can kill living cells, i.e. gamma-rays may be used to kill cancerous cells. X-rays (1895 Roentgen) are generated by bombarding a metal target with an energetic beam of electrons which can be used to obtain diagnostic information, as well as in cancer therapy and to destroy cancerous cells and tumors by damaging their DNA. Ultraviolet light (Ritter 1801) is generally generated in gasses (e.g. krypton, hydrogen), or by UV laser diodes. It is used to sterilize rooms, equipment and medical instruments. It is also used as a medical treatment. Infrared radiation (Herschel 1800) is emitted or absorbed by molecules when they change their rotational vibrational movements (IR spectroscopy). Microwave radiation (Maxwell, Hertz, Righi, Bose 1880's) used in diagnostic applications: tumor detection based on differences in tissue electrical properties. Regional hyperthermia integrated with MRI, heart and other tissue ablation. Radio waves (Hertz

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1887) – used in NMRI that uses a magnetic field and radio waves to produce detailed 3D pictures of the body organs and structures.

There is the infrared region with wavelengths longer than visible light outside the visible spectrum, at its red end. The IR region of electromagnetic radiation spans photon wavelengths from $7 \cdot 10^{-7}$ m to 10^{-3} m (400 THz to 300 GHz). The origins of IR photons are gases, liquids, and solids that make up the universe. These IR sources can both absorb and emit IR radiation. The dependence of radiation absorption and emission spectra on the wavelength identifies and provides detailed information about the materials in our universe and processes in our bodies.

Infrared radiation sensors can be generally divided into two classes:

- Photon detectors in which the radiation absorption process directly produces a measurable effect (e.g. generation of photoelectrons or charge carrier pairs in a photoconductor).
- Thermal detectors that are also considered as indirect optical detectors, as they first convert the absorbed incident radiation into heat, which then produces a measurable effect.

Thermal sensing devices have the following five main advantages over photon IR sensors:

- Sensitivity in a very large spectral bandwidth limited only by the ability of the sensor to absorb the incident radiation.
- Sensitive in a very wide temperature range without the need of cooling.
- Low power requirements.
- Relatively fast response.
- Low-cost materials.

2. PHASE TRANSITIONS IN PYROELECTRIC CRYSTALS

Since time immemorial, people in India and Ceylon, where tourmaline crystals are easily found, have been observing that such crystals, when thrown into ashes, would strongly attract surrounding ash particles after a few moments, only to repel them again a little later. Dutch merchants brought this knowledge together with the first tourmaline crystal to Europe around 1703, where tourmaline was often called “Ceylon magnet”. Carl Linnaeus gave it the scientific name “Lapis Electricus” in 1747. Aepinus proved the electrical nature of this phenomenon in 1756 when he noted the opposite polarities at two ends of a heated tourmaline crystal. Brewster named this effect “pyroelectricity” (from Greek “pyro”, which means *fire*) in 1824.

Pyroelectricity is the ability of the material to generate a temporary voltage when it is heated or cooled. The change in temperature modifies the positions of the atoms slightly within the crystal structure, in such a way that the polarization of the material changes [1, 2].

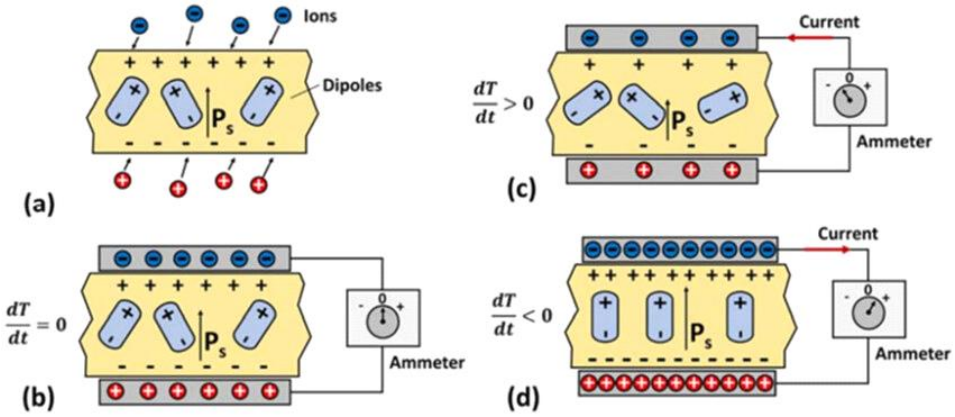


Fig. 1. Explanation of pyroelectric phenomenon. (Reproduced with permission from Kishore et al., Materials 11(8), 1433 (2018). Copyright 2018 Author(s), licensed under a Creative Commons Attribution 4.0 License); (a) Spontaneous polarization of pyroelectric materials induced by internal electric dipoles. (b) The mechanism of pyroelectric effect for the pyroelectric material under steady temperature condition. When temperature is held constant (dT/dt = 0), the internal electric field caused by spontaneous polarization in pyroelectric material and the induced external electric field in two electrodes are in equilibrium, there is no current in the steady state. (c) The mechanism of pyroelectric effect for pyroelectric material under heating condition. When temperature is increased (dT/dt > 0), the net dipole moment will be caused and spontaneous polarization will decrease, which will drive the migration of electrons in the external circuit. (d) The mechanism of pyroelectric effect for pyroelectric material under cooling condition. When temperature is decreased (dT/dt < 0), the spontaneous polarization will be enhanced and the electrical balance is broken again, which then causes reverse electron migration.

To date, many compounds/materials exhibiting pyroelectric properties have been discovered. They are widely used in active IR sensing. They belong to different material groups and some of them are listed in Tab. 1.

A phase transition is the transformation of a thermodynamic system from one phase to another by heat transfer. The term is most commonly used to describe transitions between solid, liquid and gaseous states of the matter, and, in rare cases, plasma.

First-order phase transitions are those that involve latent heat. During such a transition, the system either absorbs or releases a fixed (and typically large) amount of energy per volume. During this process, the temperature of the system will stay constant, as heat is added: the system is in a "mixed phase regime" in which some parts of the system have completed the transition and others have not. Familiar examples are melting of ice or boiling of water (water does not instantly turn into vapor, but forms a turbulent mixture of liquid water and vapor bubbles).

Tab. 1. Important pyroelectric and ferroelectric materials [3]

Ferroelectric Family	Ferroelectric Material	Chemical Formula	Abbreviation
Perovskite type	Barium (Ba) titanate	$BaTiO_3$	BTO
	Potassium (K) niobate	$KNbO_3$	KN
	K titanate	$KTiO_3$	KT
	K tantalate-niobate	$KTa_{1-x}Nb_xO_3$	KTN
	Pb zirconate titanate	$PbZrxTi_{1-x}O_3$	PZT
	Pb scandium titanate	$PbSc_xTi_{1-x}O_3$	PST
	Ba strontium (Sr) titanate	$Ba_xSr_{1-x}TiO_3$	BST
Li niobate family	Li niobate	$LiNbO_3$	LN
	Li tantalate	$LiTaO_3$	LT
Tungsten-bronze type	Ba Sr niobate	$Ba_{5x}Sr_{5(1-x)}Nb_{10}O_{30}$	BSN
	Ba sodium niobate	$Ba_{5x}Na_{5(1-x)}Nb_{10}O_{30}$	BNN
	K Li niobate	$K_5Li_2Nb_5O_{15}$	KLN
KDP family	K dihydrogen phosphate	KH_2PO_4	KDP
	K dihydrogen arsenate	KH_2AsO_4	KDA
	Rubidium dihydrogen phosphate	RbH_2PO_4	RDP
TGS type	Triglycine sulfate	$(NH_2CH_2COOH)_3{\cdot}H_2SO_4$	TGS
	Triglycine selenate	$(NH_2CH_2COOH)_3{\cdot}H_2SeO_4$	TGSe
KTP family	K titanyl phosphate	$KTiOPO_4$	KTP
Bismuth titanate	Bismuth titanate	$Bi_4Ti_3O_{12}$	BITO
Rare earth molybdate	Gadolinium molybdate	β-$Gd_2(MoO)_3$	GMO
Pb germanium oxide	Pb germanium oxide	$5PbO{\cdot}3GeO_2$ or $Pb_5Ge_3O_{11}$	LGO
Antimony sulphoiodide	Antimony sulphoiodide	SbSI	SbSI
Polymer	Poly (vinylidene fluoride)	–	PVDF
	Poly (vinylidene fluoride-trifluoroethylene)	–	P(VDF-TrFE)

Important: two phases coexist, the transformation is completed over a finite range of temperatures, hysteresis is observed on thermal cycling.

Second-order phase transitions are also called "continuous phase transitions". They are characterized by a divergent susceptibility, an infinite correlation length, and a power law decay of correlations near criticality. The examples of second-order phase transitions are the ferromagnetic transition, superconducting transition and superfluid transition. In the material referred to in this article (TGS), we are dealing with a phase transition of the second order.

Lev Landau gave a phenomenological theory of second-order phase transitions. It can be described by the equation:

$$F = F_0 + \frac{\alpha}{2}P^2 + \frac{\beta}{4}P^4 + \frac{\gamma}{6}P^6 - EP, \tag{1}$$

F – free energy density, Fo – free energy density of the paraelectric phase (when external field E = O), α, β, γ – the expansion coefficients, P – polarization (order parameter). Both free energy and polarization depend on temperature T.

Equilibrium conditions correspond to the minimum of the free energy density.

$$\frac{\partial F}{\partial P} = 0 \quad \text{and} \quad \frac{\partial^2 F}{\partial P^2} > 0. \tag{2}$$

After solving the equation the energy density F in paraelectric (T<Tc) and ferroelectric (T > Tc) phase can be visualized, as presented in Fig. 2, where Tc is the critical temperature of the phase transition.

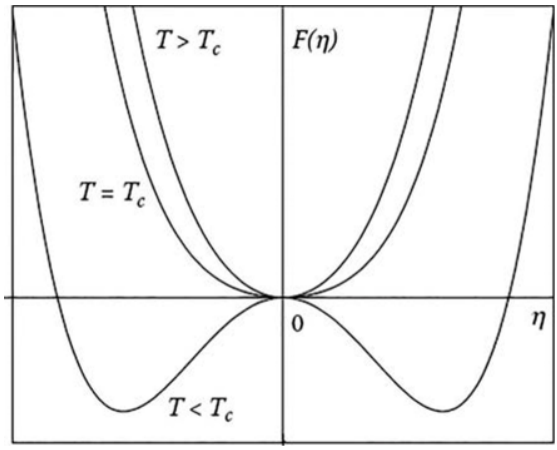


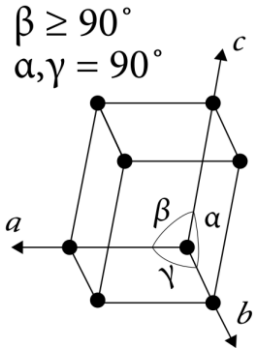
Fig. 2. Dependences F(η) near the temperature of the second-order phase transition (η- ordering parameter = P) [4]

As can be seen in the ferroelectric phase $T<T_c$, there are two stable states of thermodynamic equilibrium (potential wells). In real ferroelectrics, the occurrence of such states is related to interactions between atoms/molecules of the material. This state is called the ordered state. Once the critical temperature T_c is exceeded, thermal motions cause disruption of interatomic relationships (e.g. weak hydrogen bonds), resulting in a transition to a disordered state and reaching the paraelectric phase.

3. TRIGLYCINE SULPHATE CHARACTERIZATION

3.1. Crystal structure

Triglycine sulfate (TGS) is a hybrid organic–inorganic crystal. It is one of the most comprehensively studied ferroelectric materials for infrared, non-cooled thermal detectors (FTIR spectroscopy etc.). This is an uniaxial ferroelectric model and is attractive because of its excellent pyroelectric properties, high figures of merit and easy growing process. TGS single crystals are grown from water solutions in a controlled evaporation process. The elementary cell of a single crystal is monoclinic in both polar and non-polar phases. TGS unit cell is presented in Fig. 3. and the network of molecules configuration in paraelectric and ferroelectric phase is shown in Fig. 4.



TGS unit cell parameters:
a=9.42Å, b=12.64 Å, c=5.73 Å,
ß=110.23°

Fig. 3. Monoclinic unit cell with x, y, z coordinate axes, showing axial lengths (a, b, c), and interaxial angle together with parameters of the TGS unit cell measured in experiments

The solution for crystal growth has to be prepared according to the chemical formula:

$$3\left(NH_2CH_2COOH\right) + H_2SO_4 = \left(NH_2CH_2COOH\right)_3 \cdot H_2SO_4$$

Aminoacetic and sulphuric acids are mixed with distilled water and crystals are grown using a slow evaporation of the solvent (water) in stabilized temperature, as described in [5].

Milestones in researches of TGS:

- Ferroelectric properties of TGS discovered by Matthias, Miller and Remeik 1956.
- Description of pyroelectric phenomenon - Savage i Miller 1959.
- X-ray studies, the definition of symmetry and unit cell size - Hoshino, Okaya and Pepinsky – 1959.

- Explanation for the ferroelectric properties of TGS single crystals – Gałanow and Kisłowski 1969.
- Phase transition in triglycine family of hydrogen bonded ferroelectrics: The interpretation based on structural studies, Choudhury, 2004 [6].

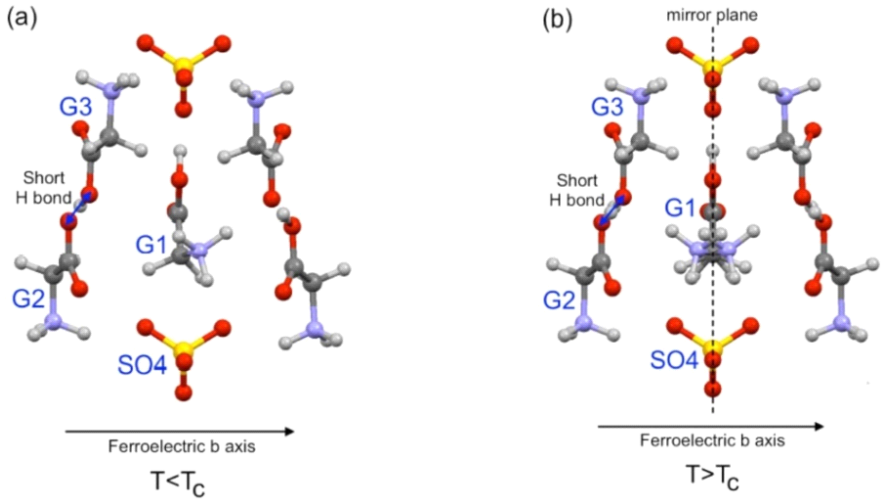


Fig. 4. Network of molecules in the unit cell of TGS in the (a) ferroelectric and (b) paraelectric phases [7]. Glycine G1 is responsible for the spontaneous polarisation and pyroelectric properties of the single-crystal TGS in the direction of the polar **b**-axis.

Theoretical models of pyroelectric phenomenon in pure and doped TGS single crystals were developed by numerous authors and their electric parameters were investigated and published in numerous research papers [7-10].

The mechanism of phase transition and the occurrence of pyroelectric effect in TGS can be presented in some simplification. The unit cell of TGS consists of three glycine groups: G1, G2, G3, and a sulphate ion in an asymmetric unit (Fig. 4.). The structures of its ferroelectric and paraelectric phases were determined with space groups being P_{21} and $P_{21/m,}$ respectively. G1 and G3 are quasi-planar and arranged nearly perpendicular to polar **b**-axis. G1 has two equivalent positions, one on the left and the other one on the right, along polar **b**-axis. For the temperature lower than T_c,, G1 occupies one of the sites with higher probability what leads to spontaneous polarization of the sample in macro scale (Fig. 4.a. and 4.b.). For the temperatures higher than critical transition temperature T_c,, G1 is disordered between equivalent sites making the polarization along the polar **b**-axis disappear (Fig. 4.b.). Disappearing polarization causes the appearance of a free charge on the samples electrodes perpendicular to the polar **b**-axis.

Many authors investigate electric parameters, such as pyroelectric coefficient, complex dielectric constant, spontaneous polarization, coercive field of the

TGS single crystals, and also its derivative forms such as composites or thin films [11-19]. The most essential parameters of TGS are as follows:

$$P_s = 0.03\ \left[\frac{C}{m^2}\right]$$
$$E_c = 16\ \left[\frac{kV}{m}\right]$$
$$\varepsilon'_{\max} = 8000_{1\text{kHz}}$$
$$\gamma = 0.02\ \left[\frac{C}{m^2 K}\right]$$

P_s – spontaneous polarization
E_c – coervive field
ε'_{max} – maximal value of electric permitivity real part
γ – piroelectric coefficient

Phase transition in TGS single crystals is also related with changes within domain structure. The ferroelectric domain is an area of oriented spontaneous polarization. Domains are seperated by domain walls. Building up a domain structure minimises internal energy of the crystal. When the TGS monocrystal is heated in the paraelectric phase (T > Tc = 322 K, 49°C), and then cooled to the ferroelectric phase, non-equilibrium domain structure appears. It is easy to notice a set of very small domains. For energy reasons such a domain structure gradually enlarges over temperature decrease, as presented in Fig. 5. The domain structure also changes with time, even when the temperature is constant. This proces is called ageing of the crystal and making the crystal to be depolarised with time.

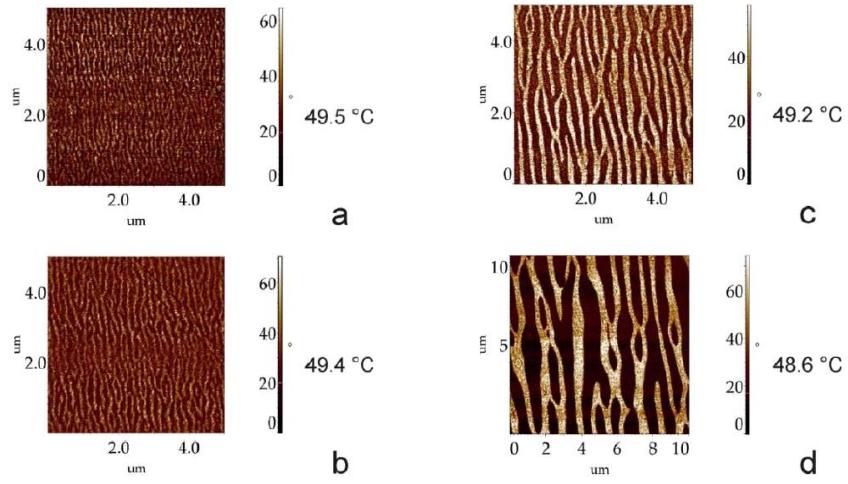


Fig. 5. Temperature evolution of the quasiperiodic polarized structure under transition through T_C in TGS crystal cooling: 49.5 °C (a), 49.4 °C (b), 49.2 °C (c), 48.6 °C (d) [9]

The domain structure and its parameters such as the size of the domains, their shape or surface density are closely related to the electrical properties of TGS single crystals.

3.2. Pyroelectric properties

As mentioned in the Pyroelectrics section, the ability of a crystal to generate temporary voltage when heated or cooled is called pyroelectricity. When the temperature is changed, the position of atoms in the crystal structure is slightly changed, resulting in a change in polarization, which later results in the generation of voltage. The pyroelectric effect can be defined as the change of polarization as a function of temperature.

Pyroelectric coefficient can be defined as:

$$\gamma = \frac{\partial P_S}{\partial T} \tag{3}$$

It can be calculated on the basis of the measurement of pyroelectric current.

$$I = \frac{dQ}{dt} = S\frac{dP_S}{dt} = \gamma S\frac{dT}{dt} \tag{4}$$

I – electric current of short circuit sample,
γ – pyroelectric coefficient,
S – samples area,
T – temperature,
t – time,
Q – electric charge,
P_s – spontaneous polarization.

For the pyroelectric materials exhibiting the second order phase transition (like TGS) the following theoretical relation between spontaneous polarization and pyroelectric current may be observed, as presented in Fig. 6.

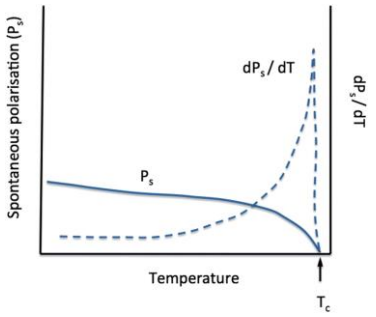


Fig. 6. Temperature dependence of spontaneous polarization Ps and pyroelectric coefficient dPs/dT of a ferroelectric material exhibiting a phase transition of the second order [20]. Tc – critical temperature of the phase transition.

Samples area can be easily measured. It is also relatively easy to keep the dT/dt rate constant (linear heating/cooling). The temperature characteristics of pyroelectric coefficient $\gamma(T)$ can be calculated from equation 4. This method, which is called static, is one of the most popular in the investigation of pyroelectric coefficient of TGS samples.

The pyroelectric effect in TGS is one of the most important phenomena for practical applications of these ferroelectric single crystals as active elements of pyroelectric sensors. Nonlinear properties of pure, doped and powdered (composite) materials based on TGS have been investigated by numerous authors [16-19]. During the research on the electric properties of TGS single crystals we have observed various dependences of the temperature characteristics of pyroelectric coefficient depending on the sample dimensions, domain structure, heating rate etc. [12, 14]. This area is still promising and important, as ferroelectrics play an essential role in contemporary electronics.

4. EXPERIMENTAL

4.1. Pyroelectric measurements

The idea of static method for the measurement of pyroelectric effect is presented in Fig. 7. Pyroelectric charge induced by temperature change dT/dt produces pyroelectric current and voltage drop measured by Digital pyrometer V. The pyrometer is connected to PC LabView software by a standard RS232 cable. The temperature of the sample fixed in the sample holder is measured by two Pt100 sensors. Pyroelectric signal and temperature signals are registered for analysis and visualization.

Temperature dependencies of pyroelectric voltage measured for TGS cubic sample in three crystallographic directions (a, b, c) are presented in Fig. 8.

Measurements of the pyroelectric properties of materials used in IR detectors are crucial in terms of determining their sensitivity and dynamic properties. The results of the studies presented above proved that the mechanism of phase transition in a TGS single crystal is three-dimensional in nature. Weak signals were observed in directions orthogonal to the polar **b**-axis, which indicates the existence of some intermolecular interactions preceding the "main" phase transition with a maximum at the critical temperature Tc.

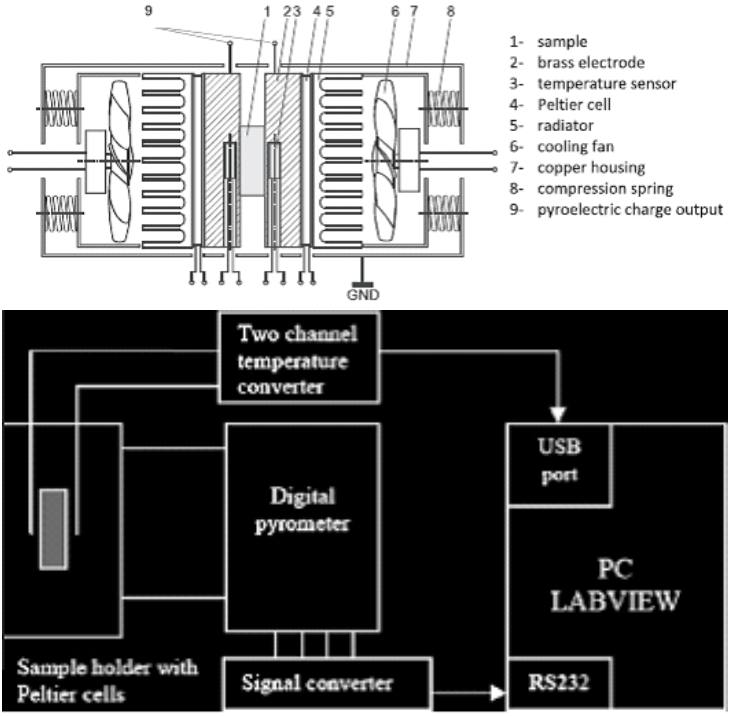


Fig. 7. Cross section of the sample holder and the idea of home-made pyroelectric signal measurement system

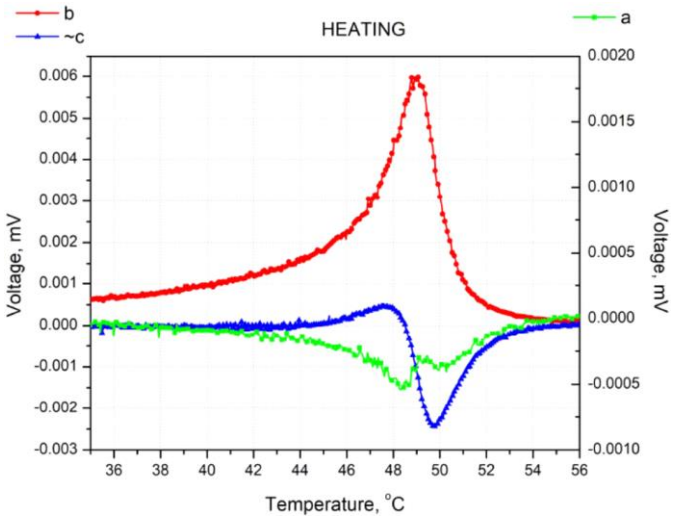


Fig. 8. Pyroelectric response of the cubic sample during heating. Red circles (crystallographic **b** – polar axis), yellow squares (crystallographic **a** axis), blue triangles (crystallographic **c** axis) [21].

4.2. Dielectric properties

The best way to determine electrical properties of a dielectric material is by broadband dielectric spectroscopy (BDS). It is a powerful technique used to study the properties of dielectric materials over a wide range of frequencies. It provides information about how a material responds to an applied electric field across a broad spectrum of frequencies, typically spanning from sub-Hertz (10^{-4} Hz) to several terahertz (10^{12} Hz). BDS is widely used in various fields such as physics, chemistry, materials science, and engineering to investigate the dynamics and behavior of materials. BDS can provide detailed information about various dielectric properties of materials and its temperature characteristics, such as:

- Complex dielectric constant – real part (permittivity) - the extent to which a material can store electrical energy in an electric field.
- Complex dielectric constant – imaginary part – dielectric loss (loss tangent) – the amount of energy lost as heat when an electric field is applied.
- Dielectric relaxation: The response of a material to an alternating electric field, which can reveal information about molecular motions and dynamics within the material.
- Complex conductivity – the ability of a material to conduct electric current, which can be frequency-dependent in materials with mobile charge carriers.

Our measurements were conducted with the use of Novocontrol ALPHA-A dielectric spectrometer presented in Fig. 9.

Fig. 9. Dielectric spectrometer Novocontrol ALFA-A used in dielectric measurements

Temperature and frequency spectrum of complex dielectric constant **ε**, measured for powdered TGS single crystal is presented in Fig. 10. [19].

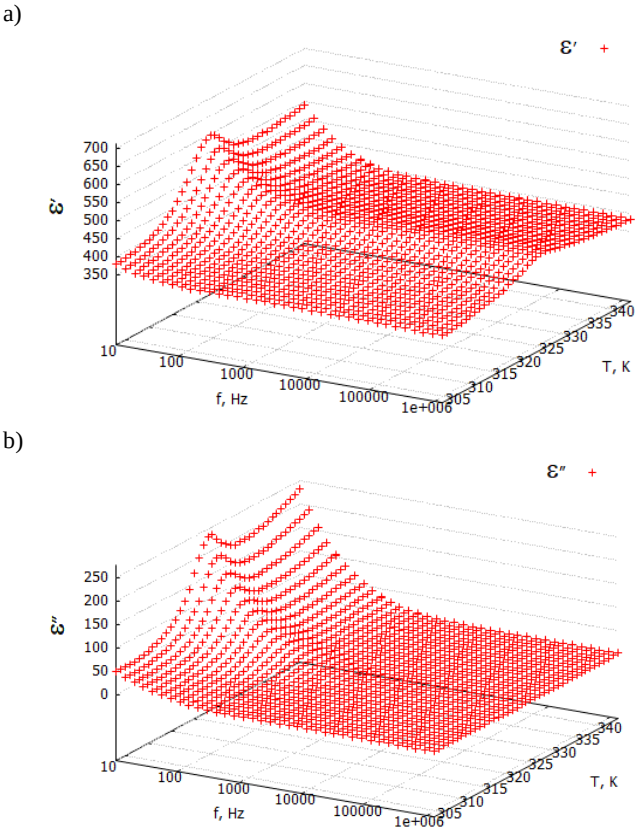


Fig. 10. Temperature and frequency spectrum of complex dielectric constant **ε**, measured for powdered TGS single crystal a) real part ε' and b) imaginary part ε''

In the study above, the samples were made of ground monocrystalline TGS. Grain gradation was about 5 μm. The characteristic peak accompanying the phase transition in the monocrystalline samples at 322 K can be observed in Fig. 10. Thus, the effect of grain size on the dielectric properties of ferroelectric materials can be studied to be used in composites.

5. CONCLUSION

The range of applications for thermal – pyroelectric detector is very wide. TGS-based pyroelectric detectors offer high sensitivity, fast response times, and wide dynamic range, making them suitable for various applications in infrared

sensing, thermal imaging, gas detection, temperature measurement, motion sensing, and energy harvesting.

TGS has garnered significant interest from specialists in both fundamental and applied research fields for nearly 70 years. As a ferroelectric model exhibiting second-order phase transition, the material facilitates fundamental research into the phase transition mechanism within this category of materials. Recently, the increasing interest in this material, driven by its possible application in the production of multifunctional composites – whether based on powders or thin films – has maintained its relevance and significance in contemporary materials engineering.

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