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DIELECTRIC PROPERTIES OF COMPOSITES BASED ON TRIGLYCINE SULFATE AND BARIUM TITANATE NANOPARTICLES

S.V. Baryshnikov[1], *A.Yu. Milinskiy*[1], *E.V. Stukova*[2]✉

[1] Blagoveshchensk State Pedagogical University, Blagoveshchensk, Russia;

[2] Amur State University, Blagoveshchensk, Russia

✉ lenast@bk.ru

Abstract. Studies of the dielectric properties of composites $(TGS)_{1-x}/(BaTiO_3)_x$ are presented ($x = 0.10$ and 0.20) obtained by mixing TGS powders with particles of 3–5 microns and $BaTiO_3$ with a particle size of 400 nm. For composite samples, an increase in the Curie temperature by $\Delta T_c \approx 3$ °C was revealed when heated for TGS, which is part of composites, an increase in the dielectric constant of composites and a decrease in the tangent of the dielectric loss angle compared with pure TGS samples. The appearance of temperature hysteresis and blurring of the TGS phase transition in the composition of composites has been detected.

Keywords: triglycine sulfate, composite, dielectric constant, nanoparticles, barium titanate, phase transition

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ДИЭЛЕКТРИЧЕСКИЕ СВОЙСТВА КОМПОЗИТОВ НА ОСНОВЕ ТРИГЛИЦИНСУЛЬФАТА И НАНОЧАСТИЦ ТИТАНАТА БАРИЯ

С. В. Барышников[1], *А. Ю. Милинский*[1], *Е. В. Стукова*[2]✉

[1] Благовещенский государственный педагогический университет, г. Благовещенск, Россия;

[2] Амурский государственный университет, г. Благовещенск, Россия

✉ lenast@bk.ru

Аннотация. Представлены исследования диэлектрических свойств композитов $(TGS)_{1-x}/(BaTiO_3)_x$ (x = 0,10 и 0,20), полученных путем смешивания порошков TGS с частицами размером 3–5 мкм и $BaTiO_3$ с размером частиц 400 нм. Для композитных образцов выявлен рост температуры Кюри на $\Delta T_c \approx 3$ °C при нагреве для TGS, входящего в состав композитов, увеличение диэлектрической проницаемости композитов и уменьшение тангенса угла диэлектрических потерь по сравнению с чистыми образцами TGS. Обнаружено появление температурного гистерезиса и размытие фазового перехода TGS в составе композитов.

Ключевые слова: триглицинсульфат, композит, диэлектрическая проницаемость, наночастицы, титанат бария, фазовый переход

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Introduction

Organic ferroelectrics with hydrogen bonds, such as ferric acid and triglycine sulfate (TGS), are important research subjects for the electronics industry due to their unique properties. One of the key characteristics of these materials is their ability to switching of spontaneous polarization, allowing to create highly efficient electronic components capable of operating in a wide range of technological applications. These ferroelectrics are used to manufacture memory chips, transistors, capacitors, etc. [1–6].

TGS is a classic well-studied ferroelectric. Recent years have seen an increase in the number of studies of composite and nanocomposite structures based on TGS. These new research directions open up prospects for the creation of materials with improved characteristics and expanded functionality [7–12]. It was found in [9] that TGS can interact with nanocellulose particles through hydrogen bonds, which leads to an increase in the Curie temperature for TGS. An increase in the Curie temperature was observed in [10] for TGS with oxidized carbon nanotubes. The formation of hydrogen bonds between TGS and Al_2O_3 was observed in [11]. The magnetoelectric composite TGS + Fe_3O_4 was considered in [12], suggesting that magnetoelectric coupling appeared for the TGS + 10 wt.% Fe_3O_4 composite, as indicated by the shift in the temperature of the phase transition in TGS.

This paper reports on the study of dielectric properties and pyroelectric current in $(TGS)_{1-x}/(BaTiO_3)_x$ composites (x = 0.10, 0.20) obtained based on TGS and tetragonal $BaTiO_3$ nanoparticles with a size of 400 nm.

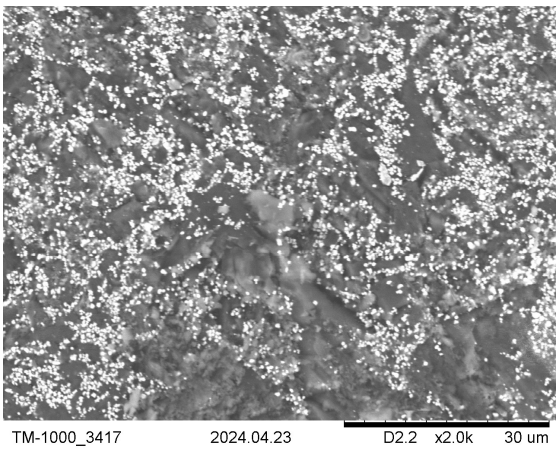

Fig. 1. Electron micrograph of $(TGS)_{0.90}/(BaTiO_3)_{0.10}$ composite surface: 400 nm $BaTiO_3$ particles, obtained at 5000× magnification

Samples and experimental procedure

TGS crystals heated to T_c = 49 °C undergo a phase transition from the ferroelectric to the paraelectric phase. TGS exhibits polar monoclinic symmetry below T_c. Spontaneous polarization is directed along the b axis, amounting to 2.8 μC/cm² at 20 °C. Above T_c, TGS is in monoclinic nonpolar phase [13].

Barium titanate ($BaTiO_3$) is in tetragonal phase with spontaneous polarization of 26 μC/cm² at room temperature. Above 120 °C, $BaTiO_3$ passes into the cubic paraphase [13]. This study used barium titanate nanopowders with an average particle size of 400 nm (MannGrainNano Technology Co., Ltd.). Their purity was 99.9%. $BaTiO_3$ particles with a size of 400 nm were characterized by tetragonal symmetry in the temperature range under study. The composite materials of the $((TGS)_{1-x}/(BaTiO_3)_x$ system were synthesized by mechanically mixing TGS powder and $BaTiO_3$ nanoparticles in an agate mortar

for 10 minutes. The TGS particles had an average size of 5 μm, and the $BaTiO_3$ particles had an average size of 400 nm. The resulting mixture was pressed at 7,500 kg/cm^2 into samples with a diameter of 10 mm and a thickness of 1 mm. In this study, we considered $(TGS)_{0.90}/(BaTiO_3)_{0.10}$ and $(TGS)_{0.80}/(BaTiO_3)_{0.20}$ composites, while compressed TGS powders were used as reference samples. Fig. 1 shows electron micrographs for the surface of $(TGS)_{0.90}/(BaTiO_3)_{0.10}$ composites.

The temperature dependences of dielectric constant of composites were studied at a frequency of 10^6 Hz using an E7-25 immittance meter. The pyroelectric effect was measured to control the effective spontaneous polarization of the samples. There are various techniques for determining the pyroelectric coefficient, measuring the voltage, charge or current occurring when the temperature of the crystal changes. However, if the total resistance of the sample changes during the temperature change, the most appropriate approach is to measure the pyroelectric current or charge under short-circuit conditions (i.e., the electric field E is maintained constant [14]). In our case, the short-circuit condition was maintained using the AD620 instrumentation amplifier. To measure the pyroelectric current, the samples were pre-polarized at room temperature and an electric field of 5000 V/cm, the heating rate during measurements was maintained constant at 5 K/min. The signal from the amplifier was fed to the computer via the ZET 210 ADC for further processing.

Experimental results and discussion

According to the $\varepsilon'(T)$ dependences for pressed TGS powder, the maximum ε' during heating and cooling is detected at about 49.5 °C (see Table). The peak value of $\varepsilon'(T)$ for the TGS sample is 45.6, which is more than an order of magnitude lower than that for a single crystal [13], due to the averaging of properties along different crystallographic axes.

Table 1

Measurement results for synthesized samples

Sample	T_h	ΔT	ε'_{max}		tgδ
			under heating	under cooling	
TGS	49.5	0.0	41.7	45.6	0.17
$(TGS)_{0.90}/(BaTiO_3)_{0.10}$	53.2	4.8	47.0	51.6	0.11
$(TGS)_{0.80}/(BaTiO_3)_{0.20}$	53.6	5.1	49.2	52.1	0.07

Notations: T_h is the temperature at which the value of ε'_{max} is observed under heating; ΔT is the hysteresis, ε'_{max} is the maximum value of dielectric permittivity; tgδ is the dielectric loss tangent at 1 kHz phase transition point.

The phase transition in $(TGS)_{1-x}/(BaTiO_3)_x$ composites is smeared, exhibiting a shift towards higher temperatures (Fig. 2). Temperature hysteresis of the phase transition is also observed. Table shows the temperatures T_h at which the maximum values of the dielectric constant are observed under heating for the studied samples, as well as the maximum values of the dielectric constant under these conditions.

The results of the pyroelectric current study for TGS polycrystal and $(TGS)_{1-x}/(BaTiO_3)_x$ composites are shown in Fig. 3. It should be noted that samples of the same size, with the same polarization conditions and heating rate were used to study the pyroelectric current. According to the results, an increase in the volume concentration of $BaTiO_3$ particles in the composite leads to an enhanced pyroelectric response. The 400 nm particles are in tetragonal phase, which is characterized by spontaneous polarization and, consequently, pronounced pyroelectric properties. Tetragonal particles of $BaTiO_3$ contribute to additional polarization of the composite due to spontaneous polarization that is an order of magnitude greater compared to TGS. An increase in the concentration of $BaTiO_3$ particles in the $(TGS)_{1-x}/(BaTiO_3)_x$ composite from 10% to 20% enhances the overall pyroelectric response of the composite by 21% (Fig. 3).

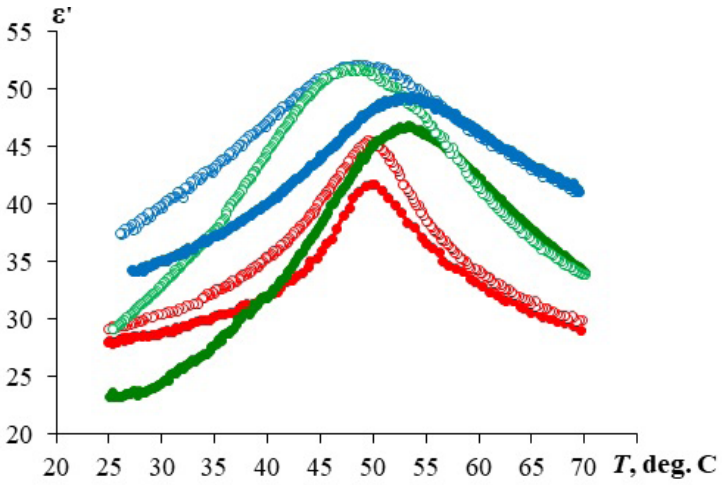

Fig. 2. Dependences $\varepsilon'(T)$ for TGS (red symbols) and $(TGS)_{1-x}/(BaTiO_3)_x$ composites (green symbols correspond to $x = 0.10$, blue symbols correspond to $x = 0.20$)
Data were obtained for heating and for cooling (shaded and empty symbols, respectively)

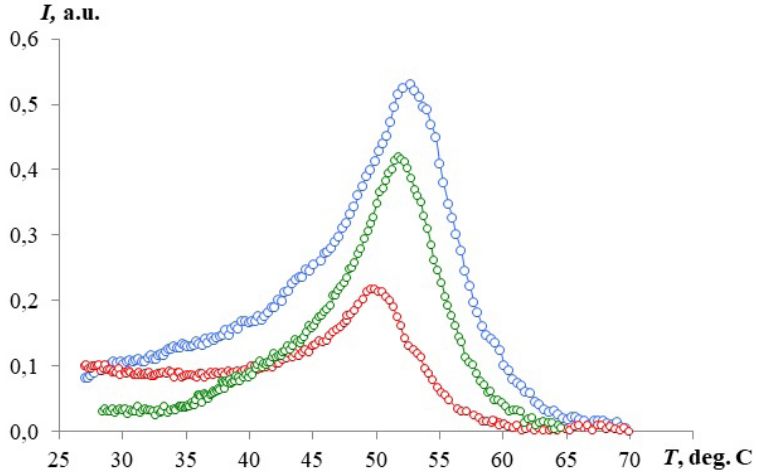

Fig. 3. Pyroelectric current for TGS samples (red symbols) and $(TGS)_{1-x}/(BaTiO_3)_x$ composites for $x = 0.10$ (green symbols) and 0.20 (blue symbols)
Data were obtained for samples of the same size,
with the same pre-polarization conditions and heating rate

According to the literature, the main causes of the Curie temperature shift in ferroelectric composites are electrical interactions, although other factors such as mechanical stresses [15] or the formation of hydrogen bonds between composite components [11, 16] are not excluded.

The main conclusions of this study are the smearing of the ferroelectric phase transition, an increase in the Curie temperature, and the appearance of temperature hysteresis for TGS particles included in the composites.

The smearing of the ferroelectric transition in TGS can be explained within the framework of the theory of phase transitions developed by Landau and Ginzburg. To account for inhomogeneous polarization, an additional term is introduced into the Landau–Ginzburg expansion, which includes the first derivatives of polarization with respect to the coordinates $(\nabla P)^2$ [17]:

$$F = \frac{1}{2}\alpha_o(T - T_o)P^2 + \frac{1}{4}\beta P^4 + \frac{1}{2}\delta(\nabla P)^2 - EP, \quad (1)$$

where α_o, β, δ are temperature-independent coefficients. Expansion (1) was used in [18] to obtain the equation defining the $\varepsilon'(T)$ dependence taking into account the inhomogeneous distribution of polarization:

$$\varepsilon'(T) \approx 1 + \frac{1}{\alpha_o (T - T_o) + f(P(\mathbf{r}, T))}, \tag{2}$$

where $f(P(\mathbf{r}),T) = \dfrac{2\delta(T)\, d(\Delta P(\mathbf{r}),T)}{dP}$.

The presence of an additional inhomogeneous term in expression (2) leads to smearing of the $\varepsilon'(T)$ dependence obtained for an inhomogeneous ferroelectric system. The degree of this smearing is determined by the degree of inhomogeneity of the composite.

To understand the change in the Curie temperature of TGS in the $(TGS)_{1-x}/(BaTiO_3)_x$ composite, we write F of the composite as a sum of three terms, energy of the system of TGS and $BaTiO_3$ particles and the energy of their interaction. We assume the dipole-dipole interaction with the energy W_{dd} to be the main interaction of polar particles.

$$\begin{aligned} F = &\sum_i \left[\frac{1}{2}\alpha_i P_i^2 + \frac{1}{4}\beta_i P_i^4 + \frac{1}{2}\delta_i \left(\nabla P_i\right)^2 \right] + \\ &+ \sum_j \left[\frac{1}{2}\alpha_j P_j^2 + \frac{1}{4}\beta_j P_j^4 + \frac{1}{6}\gamma_j P_j^6 + \frac{1}{2}\delta_j \left(\nabla P_j\right)^2 \right]_i + \\ &+ \sum_{i,j} \left[\frac{\mathbf{p}_i^* \mathbf{p}_j^*}{r_{ij}^3} - \frac{3\left(\mathbf{r}_{ij}\mathbf{p}_i^*\right)\left(\mathbf{r}_{ij}\mathbf{p}_j^*\right)}{r_{ij}^5} \right], \end{aligned} \tag{3}$$

Taking into account the interaction energy W_{dd} in relation (3) leads to a change in the temperature of the phase transition [19].

$$\tilde{T}_o = T_o + \frac{1}{\alpha_o} W_{dd} = T_o + \frac{1}{\alpha_o} \sum_i \mathbf{p}_i^* \mathbf{E}_i^*, \tag{4}$$

where $\mathbf{p}_i^*$ are the effective values of dipole moments, and $\mathbf{E}_i^*$ are the effective local fields acting on the particle [20]. The sign of the energy W_{dd} is determined by the orientation of dipole moments of the particles. However, since the temperature of the TGS phase transition in composites increases, $W_{dd} > 0$ and the dipole moments of $BaTiO_3$ particles must be oriented so as to compensate for the field of TGS particles.

Phase transitions in ferroelectric materials are an important object of research in the field of materials science and solid state physics. From a thermodynamic standpoint, the first-order phase transition differs from the second-order phase transition by abrupt changes in the order parameter and the presence of latent heat. However, in real inhomogeneous systems such as ferroelectric materials with defects, impurities, and grain boundaries, a second-order phase transition can become a first-order phase transition. The reason for the change in the type of TGS phase transition in composites is that the energy of the dipole-dipole interaction W_{dd} has different values above and below the Curie temperature. Below T_c, TGS and $BaTiO_3$ particles interact with complete dipole moments (Keesom energy), while above that temperature, the dipole moments of $BaTiO_3$ particles in the ferroelectric phase interact with induced dipole moments in TGS (Debye energy). A change in thermodynamic potential by a different value below and above the phase transition causes an energy jump, which leads to hysteresis, and therefore the phase transition becomes a first-order transition.

Conclusion

Studies of dielectric properties of composites $(TGS)_{1-x}/(BaTiO_3)_x$ ($x = 0.10$ and 0.20) obtained by mixing TGS powders with particles of 3-5 microns and $BaTiO_3$ with a particle size of 400 nm showed the following changes in properties:

increase in the Curie temperature by $\Delta T_c \approx 3$ °C under heating for TGS included in the composites;

increase in the dielectric constant of composites, decrease in the dielectric loss tangent compared with pure TGS samples;

appearance of temperature hysteresis and smearing of TGS phase transition in composites. The hysteresis increases with an increase in the content of $BaTiO_3$ particles, amounting to $\Delta T \approx 4.8$ °C at $x = 0.10$ and $\Delta T \approx 5.1$ °C at $x = 0.20$, respectively.

We established that all the results obtained for $(TGS)_{1-x}/(BaTiO_3)_x$, i.e., the Curie temperature shift, the increase in temperature hysteresis, and the phase transition smearing, can be explained within the framework of the phenomenological Landau–Ginzburg theory, taking into account the electrical interactions between the components of composite systems and the inhomogeneity of electric fields.

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THE AUTHORS

BARYSHNIKOV Sergey V.
Blagoveshchensk State Pedagogical University
104, Lenina St., Blagoveshchensk, Russia, 675000
svbar2003@list.ru
ORCID: 0000-0002-3362-8975

MILINSKIY Alexey Yu.
Blagoveshchensk State Pedagogical University
104, Lenina St., Blagoveshchensk, Russia, 675000
a.milinskiy@mail.ru
ORCID: 0000-0001-7525-4396

STUKOVA Elena V.
Amur State University
21, Ignatievskoe shosse, Blagoveshchensk, Russia, 675027
lenast@bk.ru
ORCID: 0000-0002-7981-7456

LVOV Andrey E.
Blagoveshchensk State Pedagogical University
104 Lenin St., Blagoveshchensk, 675000, Russia
andrei.1998.lvov@gmail.com
ORCID: 0009-0004-6095-9026

СВЕДЕНИЯ ОБ АВТОРАХ

БАРЫШНИКОВ Сергей Васильевич – *доктор физико-математических наук, профессор кафедры физического и математического образования Благовещенского государственного педагогического университета.*
675000, г. Благовещенск, ул. Ленина, д. 104
svbar2003@list.ru
ORCID: 0000-0002-3362-8975

МИЛИНСКИЙ Алексей Юрьевич – *доктор физико-математических наук, профессор кафедры физического и математического образования Благовещенского государственного педагогического университета.*
675000, г. Благовещенск, ул. Ленина, д. 104
a.milinskiy@mail.ru
ORCID: 0000-0001-7525-4396

СТУКОВА Елена Владимировна – *доктор физико-математических наук, профессор кафедры физики Амурского государственного университета.*
675027, Россия, г. Благовещенск, Игнатьевское шоссе, 21
lenast@bk.ru
ORCID: 0000-0002-7981-7456

ЛЬВОВ Андрей Эдуардович – *аспирант кафедры физического и математического образования Благовещенского государственного педагогического университета.*
675000, Россия, г. Благовещенск, ул. Ленина, 104
andrei.1998.lvov@gmail.com
ORCID: 0009-0004-6095-9026

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