PREPARATION AND PHYSICAL PROPERTIES OF THE PIEZOELECTRIC COMPOSITES BASED ON $Sn_2P_2S_6$ MATERIAL

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Preparation technology of piezoelectric composites formed as the tablets under the pressure, temperature and electrical polarizing field on the base of $Sn_2P_2S_6$ material is presented. Also frequency dependencies of Z (impedance), φ (phase), R (resistance), X (reactance), C (capacity), D(dissipation factor), ε' (real part of permittivity), and ε" (imaginary part of permittivity) were investigated. It was determined that at frequencies 151,0 kHz and 739,6 kHz "damped" resonance is observed. Parameters of the piezoelectric tablet at frequency 1kHz are following: Z^S(specific)$\approx 1.92\cdot 10^6 \Omega\cdot m$, $\varphi \approx 1.513$rad, R^S(specific)$\approx 2.91\cdot 10^7 \Omega\cdot m$, X^S(specific)$\approx 1.93\cdot 10^6 \Omega\cdot m$, $C \approx 2.1\cdot 10^{-11}$F, $D \approx 0.057$, $\varepsilon' \approx 38$, $\varepsilon'' \approx 2$. Piezoelectric coefficients $d_{33} \approx 12$pC/N; $d_h \approx 119$pC/N; $g_h \approx 363\cdot 10^{-3}$V·m/N were determined too. By dynamic method, dependence of d_h from p was investigated in the range of pressure from 0.1MPa to 70MPa and at different fixed temperatures from 0^0C to 50°C. Linear increasing of value d_h with pressure was observed. Also, increasing of d_h and $d(d_h)/dp$ coefficient with increasing temperature was exposed.

1. Introduction

It's known [1-3], that $Sn_2P_2S_6$ ferroelectric monocrystals belong to monocline symmetry. In these crystals, at temperature $T_0=(339\pm3)$K there is second-order transition close to tricritical point with change of the symmetry $P_c=P2_{1/C}$. $Sn_2P_2S_6$ monocrystals have high piezoelectric performance criteria [4;5]. For example, hydrostatic piezoelectric coefficient d_h amounts to more than $300\cdot 10^{-12}$ C/N. Hydrostatic voltage coefficient g_h, determined according to the equation

$$g_h = d_h / \varepsilon \cdot \varepsilon_o \,, \qquad (1)$$

equals to more than $100\cdot 10^{-3}$ Vm/N. Relative dielectric permittivity of the sample(ε) defined by the equation

$$\varepsilon = C \cdot t / A \cdot \varepsilon_o \,, \qquad (2)$$

where *C* is electrical capacity; *t* is thickness; *A* is electrode's area of the investigated sample; $\varepsilon_0=8.85\cdot 10^{-12}$ F/m is vacuum permittivity. Value of ε of the sensitive $Sn_2P_2S_6$ monocrystal element measured at frequency 1 kHz, is about 300. That allows us to use such monocrystals for elaboration and construction of arrangements, that measure small deformations by electrical methods (piezoelectric sound sensors, seismographs) and also arrangements for converting electrical oscillations in mechanics, for example in ultrasound transmitters.

Production of big monocrystals and making piezoelectric elements from them is difficult and intricate problem. Therefore, it's essential to investigate production of piezoelectric composites on the base of $Sn_2P_2S_6$ and to analyze their properties. This paper is devoted to this problem.

2. Preparation of composites

For preparation of $Sn_2P_2S_6$ composites polycrystalline material $Sn_2P_2S_6$ was synthesized [6]. Then it was turned into the fine-grained powder in the porcelain mortar. After that, polycrystalline mass $Sn_2P_2S_6$ was riddled in the sieve, whereupon calibrated powder was received.

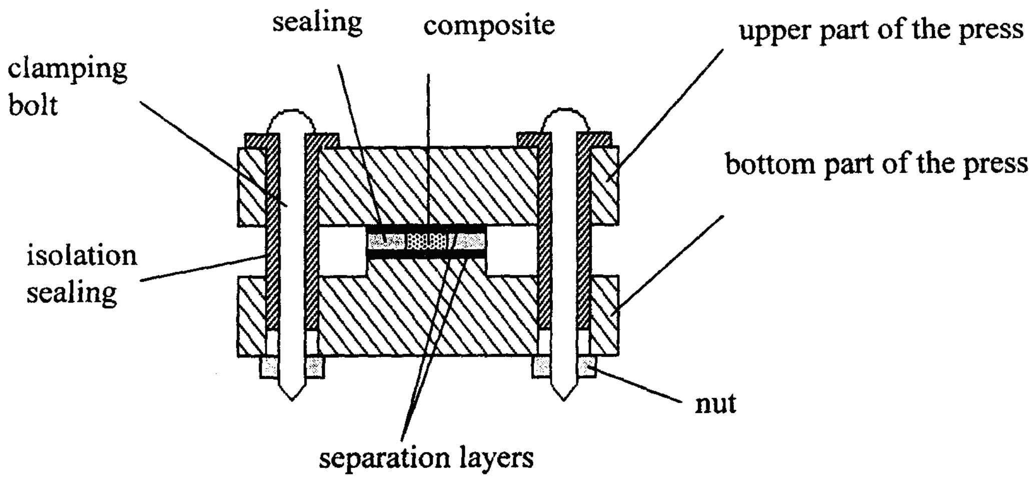


Fig. 1 Press-form for preparation of composites

First by ordinary methods using epoxy resin and hardener piezoelectric tablets with diameter 12mm and thickness 2mm were made. Electrodes produced from Ag paste Degussa were applied on the principal planes of the tablets. These tablets were polarized in the electric field E=108 V/mm at temperature T=70°C over 15 minutes, then samples were cooled down to the room temperature in the same field. Unfortunately, investigations of piezoelectric properties of these tablets indicated that we didn't obtain epoxy tablets with effective piezoelectric performance criteria by this way (table 1,n.61).

Then a method of preparation of samples in press-form under mechanical stress was developed. For this method a special press-form was made (see fig.1). It consists of two (upper and bottom) parts, which are connected by 2 screws isolated form these parts by isolation sealing.

For preparation of pressed samples a homogenous mixture from $Sn_2P_2S_6$ powder and binder in ratio 5:1 was prepared. This powder mixture was filled into a circular sealing with internal diameter 10mm and thickness 3.4mm which was placed on an inside plane of the bottom part of the press. Before this, we put a thin aluminum circle with linked Degussa under the sealing. The same circle was put over the sealing. Then the upper part of the press-form was put on the sealing and both parts were connected and tightened by clamping bolts.

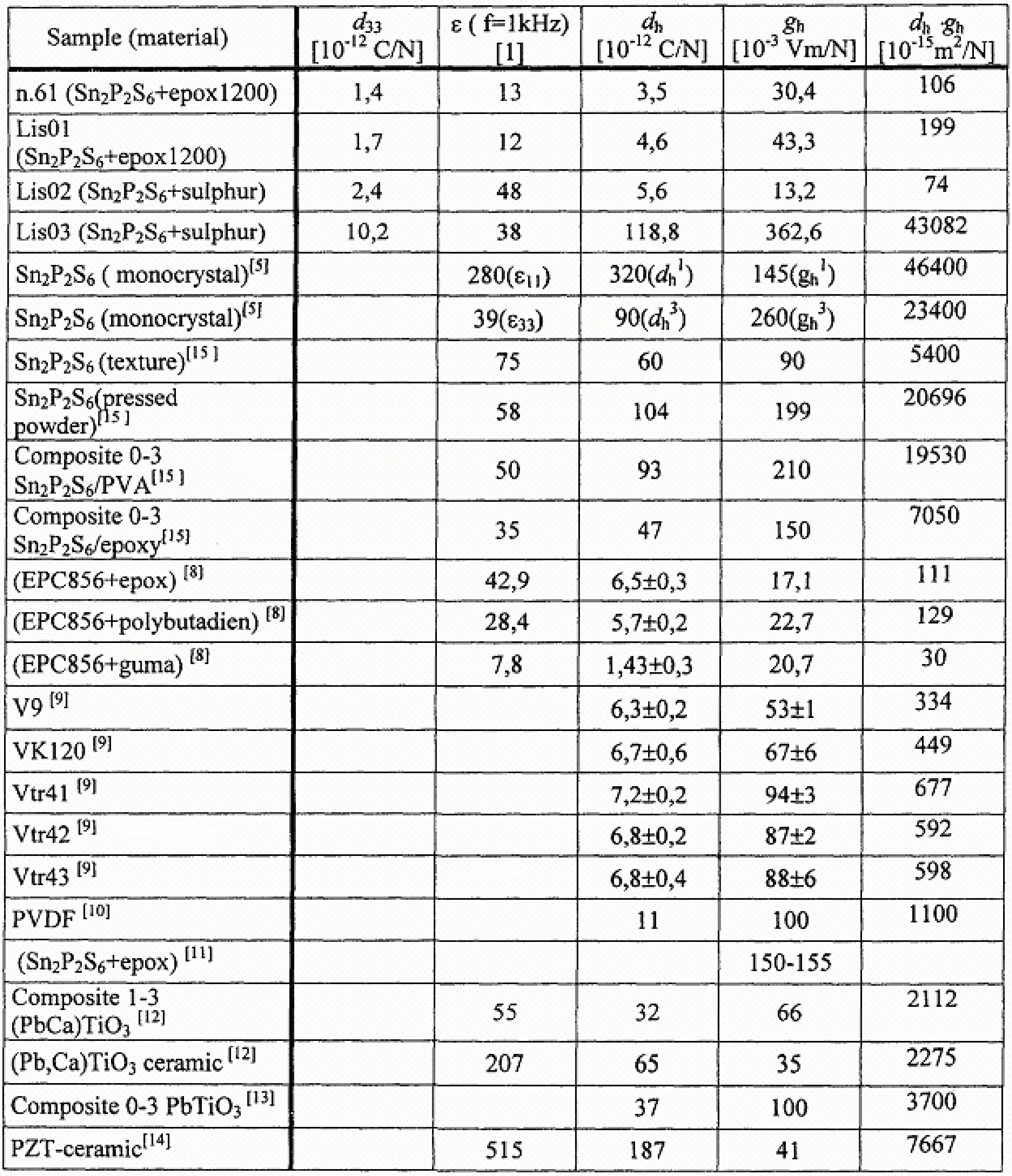
After that press-form was placed under the press and it was exposed to the determinate pressure. Compressed press-form was hold by bolts. After a time press-form with pressed sample was placed in the oil tub, in which heating, polarization and cooling were made. Press-form and tub allowed us to work at temperature range from 20°C to 200°C with applied electrical voltage on the sample up to 302 V.

Polarization of the sample passed in the following way. The sample was heated to the temperature of about 140°C. Then the voltage nearly 6V was applied on the sample. Over cooling of the sample, the voltage gradually increased and at the temperature 77°C the voltage 302V was established on the sample. At this voltage the sample was cooled down to the temperature of (32-37)°C. Then the voltage was turned off, the sample was short-circuited and left in the temperature tub for 10-20 hours for cooling down to the room temperature and for stabilizing of the physical properties. After that, the sample was taken out from the tub and from the press-form and prepared for measurements.

Resistance – temperature graph of the piezoelectric $Sn_2P_2S_6$composite sample during its polarization is shown on the fig.2.

As indicated on the fig.2, resistance of the

Table 1. Materials parameters



Sample (material)	d_{33} [10^{-12} C/N]	ε (f=1kHz) [1]	d_h [10^{-12} C/N]	g_h [10^{-3} Vm/N]	$d_h \cdot g_h$ [10^{-15}m^2/N]
n.61 ($Sn_2P_2S_6$+epox1200)	1,4	13	3,5	30,4	106
Lis01 ($Sn_2P_2S_6$+epox1200)	1,7	12	4,6	43,3	199
Lis02 ($Sn_2P_2S_6$+sulphur)	2,4	48	5,6	13,2	74
Lis03 ($Sn_2P_2S_6$+sulphur)	10,2	38	118,8	362,6	43082
$Sn_2P_2S_6$ (monocrystal)[5]		280(ε_{11})	320(d_h^1)	145(g_h^1)	46400
$Sn_2P_2S_6$ (monocrystal)[5]		39(ε_{33})	90(d_h^3)	260(g_h^3)	23400
$Sn_2P_2S_6$ (texture)[15]		75	60	90	5400
$Sn_2P_2S_6$(pressed powder)[15]		58	104	199	20696
Composite 0-3 $Sn_2P_2S_6$/PVA[15]		50	93	210	19530
Composite 0-3 $Sn_2P_2S_6$/epoxy[15]		35	47	150	7050
(EPC856+epox) [8]		42,9	6,5±0,3	17,1	111
(EPC856+polybutadien) [8]		28,4	5,7±0,2	22,7	129
(EPC856+guma) [8]		7,8	1,43±0,3	20,7	30
V9 [9]			6,3±0,2	53±1	334
VK120 [9]			6,7±0,6	67±6	449
Vtr41 [9]			7,2±0,2	94±3	677
Vtr42 [9]			6,8±0,2	87±2	592
Vtr43 [9]			6,8±0,4	88±6	598
PVDF [10]			11	100	1100
($Sn_2P_2S_6$+epox) [11]				150-155	
Composite 1-3 (PbCa)TiO_3 [12]		55	32	66	2112
(Pb,Ca)TiO_3 ceramic [12]		207	65	35	2275
Composite 0-3 $PbTiO_3$ [13]			37	100	3700
PZT-ceramic[14]		515	187	41	7667

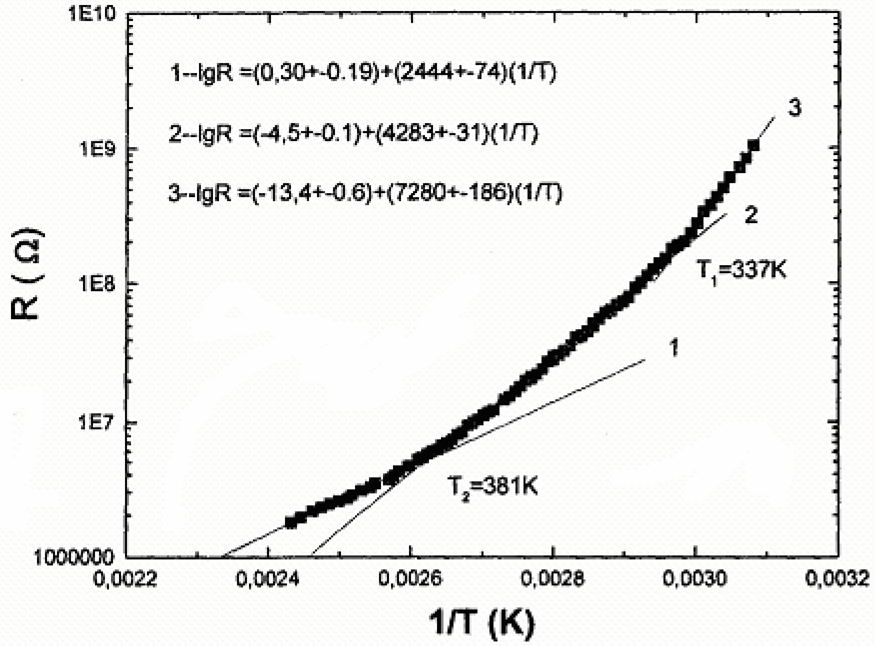


Fig.2. Temperature dependence of the electrical resistance (R) of the piezoelectric composite $Sn_2P_2S_6$ over its polarization (sample Lis 03, cooling).

sample greatly increases with cooling, at the temperatures nearly 381K and 337K in dependence R(1/T) there are kinks and at the room temperature it increased up to $3\cdot10^{11}\Omega$.

Temperature constant of the composite was approximately 7300K. Volume ratio of the $Sn_2P_2S_6$ material in pressed tablets was (65-75)%.

In such a way, using special press-form, polarized tablets of the piezoelectric $Sn_2P_2S_6$ composite with diameter of about 10mm and thickness of about (1-1.5) mm with electrodes on the principal planes made from the Degussa paste were obtained.

3. Measuring devices

For polarization of the sample, heater *Heidolph MR 3001* was used. It was supplied with the temperature tester *Heidolph EKT 3001*.Also source of the voltage *Statron Power Supply 3241.5* was used. Measurements of the current, running through the sample during the polarization, were realized by multimeter True *SMS Digital Multimeter DM-441B (EZ Digital Co.)*.

Impedance analyzer HP4192A performed measurements of the impedance characteristics of the piezoelectric samples.

Piezo d_{33}-meter ZJ-3C model (Institute of Acoustics Academia Sinsica) realized direct measurements of the piezoelectric coefficient d_{33}.

For investigations of the sample under the pressure the high-pressure chamber was worked out. Scheme of the high-pressure chamber is given on the fig.3.

It consists of corpus, cover and massive sealing head. Cover serves for leading of electric contacts to the chamber and for covering of the working space of the chamber. Circuit scheme for measurement of the samples' physical properties under the pressure is on the fig.4.

Heating element, thermocouple and investigated sample were placed into the high-pressure chamber. Pressure in the chamber was realized by silicon oil, using mechanical pump. Temperature in the chamber was regulated by thermocouple. Pressure in chamber was measured by tensometric pressure sensor TMG 760 H3G (Cresto), which was connected with the chamber by high-pressure resistant tube. For electric charge and temperature measurements on the

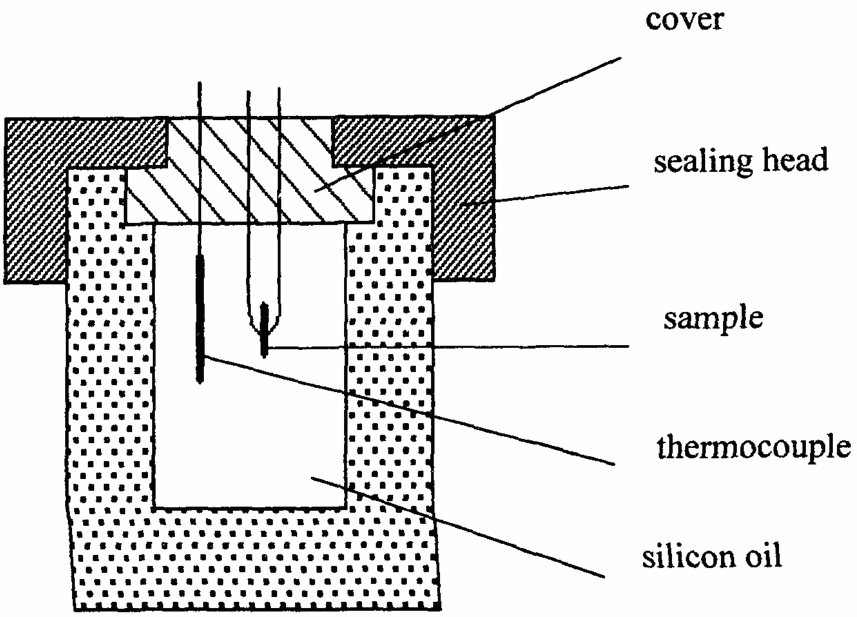


Fig. 3 Scheme of the high pressure chamber.

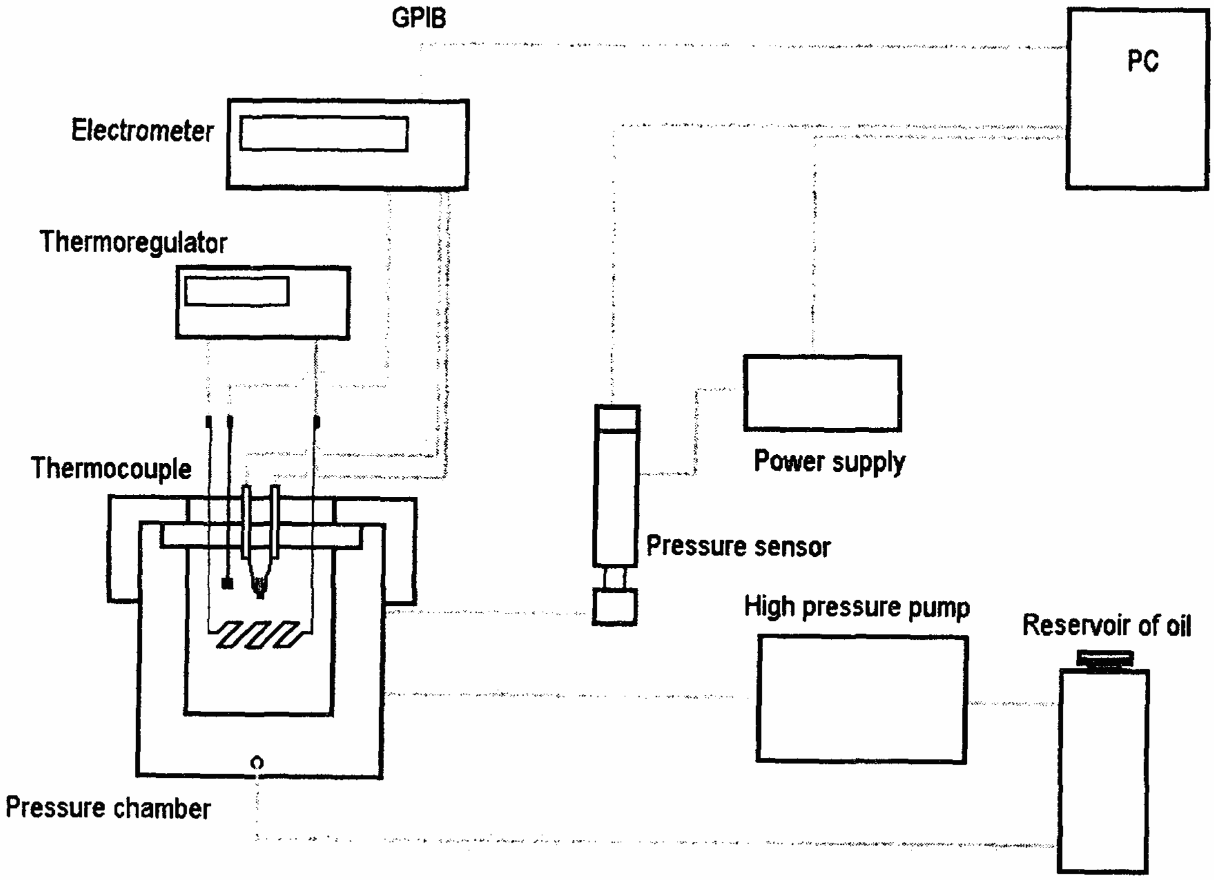


Fig.4. Circuit scheme for measurement of the physical properties of the sample under the pressure.

investigated sample, electrometer Keithley Model 6517 was used. Measurements were controlled by HPVEE program and results were registered on PC.

4. Measurements of the impedance properties

Electromechanical coupling factor (k) is an important characteristic of the piezoelectric sample. It characterizes electromechanical transformation of energies of the piezoelectric elements and it defined by the equation:

$$k^2 = U_{mech} / U_{el} \, , \qquad (3)$$

where U_{el} is electrical energy supplied on the sample; U_{mech} is mechanical energy taken from the sample. If there is transformation of energies accompanied with i-th component of an electric field and j-th component of a deformation tensor, then we can formulate:

$$k = d_{ij} / (\varepsilon_i^T \cdot s_{jj}^E / 4\pi)^{0.5} \qquad , (4)$$

where d_{ij} is piezoelectric coefficient, which describes piezoelectric oscillations, ε_i^T is a permittivity of stess free sample and s_{jj}^E is effective coefficient of an elastic compliance in constant electric field.

In such a way, using electromechanical coupling factor it's possible to compare piezoelectric materials, which have different permittivities and elastic constants.

If we apply variable potential difference to the piezoelectric element, the mechanical oscillations will arise as a result of the inverse piezoelectric effect. Amplitude of these oscillations will increase up to maximum, when the frequency of the applied variable voltage coincides with the natural frequency of the mechanical oscillations of the element. That sort of piezoelectric resonator is equivalent to oscillatory circuit, which consists of series resistance R, inductance L, capacity C1 in the first branch and capacity C2 in parallel branch.

Resonator as well as an oscillatory circuit has two resonance frequencies: f_R is series resonance frequency or inverse resonance frequency (at Z=0, where Z is complex impedance of the resonator); and f_A is parallel resonance frequency or voltage resonance frequency (at $Z=\infty$); f_R is called resonance frequency, f_A is called antiresonance frequency. They are determined by dimensions, density, and elastic, dielectric and piezoelectric properties of the sample. These frequencies are connected with electromechanical coupling factor as follows:

$$\frac{f_A - f_R}{f_R} = \frac{4}{\pi} \frac{k^2}{1-k} \, , \qquad (5)$$

then:

$$k = \frac{\pi}{2} \sqrt{\frac{\Delta f}{f_R}} \, , \qquad (6)$$

where $\Delta f = f_A - f_R$. Consequently, measurements of f_A and f_R allows us to determine an electromechanical coupling factor, which in accordance to (4), relates to piezoelectric, dielectric and elastic properties of the sample.

If the bar-shaped resonator performs the vibrations in the direction of its length, then its own resonance is determined by the equation:

$$f_R = \frac{n}{2l} \sqrt{\frac{1}{\rho \cdot s_{11}^{|}}} \, , \qquad (7)$$

where n is an index of harmonic; l is a length of the bar; ρ is density; $s_{11}^{|}$ is elastic compliance in direction of the length of the sample.

Resonator, performing vibrations in the direction of thickness of the sample usually has the shape of circular or square plate, with the thickness small as compared with diameter. The resonance frequency of this plate is determined by the equation:

$$f_R = \frac{n}{2t} \sqrt{\frac{C_{33}}{\rho}} \, , \qquad (8)$$

where t is thickness; ρ is density; C_{33} is an elastic stiffness in the direction of thickness of the plate. We investigated frequency dependencies of impedance Z, phase φ, resistance R, reactance X, capacity C and tangent of dielectric losses D. Frequency dependencies of real and imaginary part of the permittivity ε' and ε" were determined too. Results of this investigations are on

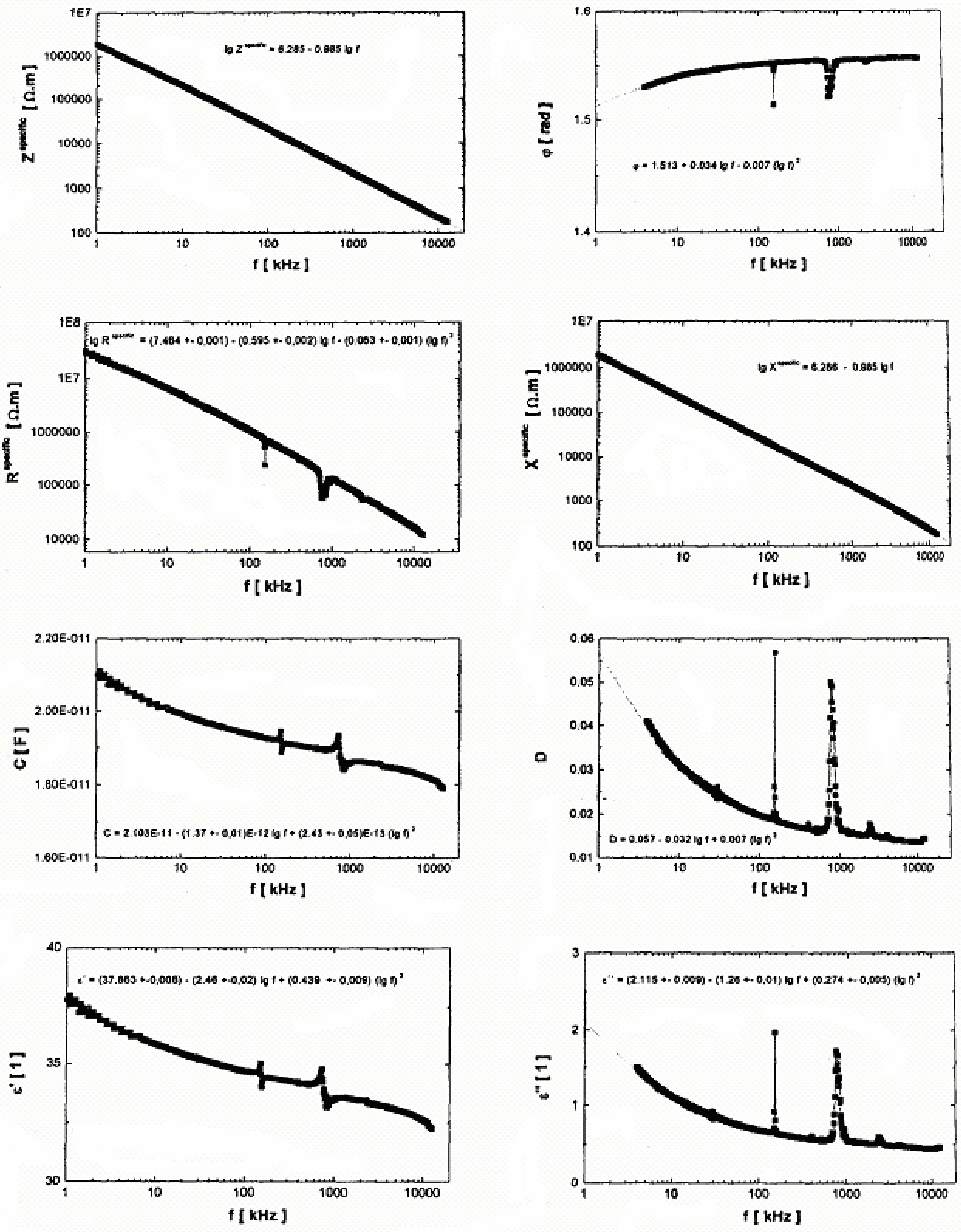


Fig. 5. Frequency dependencies of absolute impedance *Z*, phase φ, active ohmic resistance *R*, reactive resistance *X*, capacity *C*, tangent of angle of dielectric losses *D*, real part of permittivity ε', imaginary part of permittivity ε'' for a sample of piezoelectric composite of $Sn_2P_2S_6$ (disk-shaped sample Lis03, diameter 10,8 mm, thickness 1,46 mm).

fig.5. As indicated on the fig.5, Z(f) and X(f) in logarithmic scale linearly decrease with the increasing frequency. So, it's possible to express them in the following linear equations:

$$\lg Z = a_0 - a_1 \lg f \qquad (9)$$

$$\lg X = b_0 - b_1 \lg f \qquad (10)$$

where a_0 is a constant, which characterizes complex impedance of the sample; b_0 is a constant, which characterizes reactance of the sample at lgf=0. Equations of linear and quadratic approximations of experimental results Z (f), φ (f), R (f), X (f), C (f), D (f), ε'(f), ε"(f) are given on the fig.5. Dependencies Z (f) and X (f) have insignificant anomalies in the interval of frequency (151-155)kHz and (740-830)kHz. Phase φ insignificantly increases with increasing of frequency, and at the same frequencies there are clear anomalies, and this is shown by decrease of the phase that goes to zero here. But, as indicated on the fig.5, zero isn't obtained and classical piezoelectric resonance doesn't appear. Only "damped" piezoelectric resonance is observed. That is the evidence for low electromechanical coupling factor here. From the fig.5 it's seen that increasing of the frequency (f) brings to smooth decrease of the physical values (R), (D), (ε'), (ε"), which at frequencies (151-155) kHz and (740-830) kHz also have anomalies that testify about "damped" piezoelectric resonance in investigated sample. From the research results it's seen that in tablets of piezoelectric $Sn_2P_2S_6$ composite, classical piezoelectric resonance and antiresonance isn't observed. Assume, that in the interval of frequency (151-155) kHz the anomaly is caused by the radial oscillations and in the interval of frequency (740-830) kHz it's caused by the thickness oscillations of the piezoelectric tablet. Let's accept, that the beginning of the anomaly at 151kHz and 740kHz responds to the resonance (f_R) and the ending of the anomaly at 155kHz and 830khz responds to the antiresonance (f_A). After correlations (6) and (8) values of (k) and C_{33} were calculated. Our calculations have shown that coefficient k for radial and thickness oscillations equals to 0,25 and 0,55 respectively. Elastic coefficient C_{33} was calculated after the following relation:

$$C_{33} = f_R^2 \cdot 4t^2 \cdot \rho / n^2 \qquad (11)$$

For radial oscillations $C_{12} = 0{,}05 \cdot 10^{10}$ N/m^2. For thickness oscillations $C_{33} = 1{,}2 \cdot 10^{10}$ N/m^2. Then, after relation (4), piezoelectric coefficient d_{33} was calculated. For thickness oscillations $d_{33} = 92 \cdot 10^{-12}$ C/N, that is approximately 10 times higher than our experimental result ($d_{33} = 10 \cdot 10^{-12}$C/N). This disagreement appears because of classical resonance isn't observed (fig.5) and f_R and f_A were determined conditionally.

5. Investigations of the piezoelectric coefficients

Magnitudes of piezoelectric coefficient were obtained by direct measurements. Magnitudes of hydrostatic coefficient d_h were determined by measurement of the charge, which appeared on surfaces of the sample under a hydrostatic pressure [8]. Dependence of the charge density on pressure was represented by linear equation. Its gradient determines the hydrostatic piezoelectric coefficient d_h. Hydrostatic voltage piezoelectric coefficient g_h is determined by equation (1). Permittivity ε' is determined from equation (2) as a result of measurement of capacity. Results of the measurement are in table 1, compared with the values for other materials, taken from literature. From table 1 follows, that magnitude of parameter g_h is the highest for piezoelectric $Sn_2P_2S_6$ composites.

We also investigated aging processes of the piezoelectric $Sn_2P_2S_6$. composite. For that, during 200 days from the moment of production, piezoelectric coefficient d_{33} and physical values Z, R, X, D, C, ε', ε" were measured at the permanent frequency 1 kHz. Measured results showed, that for 200 days parameter d_{33} almost didn't change and had magnitude up to (10-12) pC/N. Physical values Z, R, X, insignificantly linearly increased with time. Capacity (C), permittivity (ε') slightly linearly decreased.

By dynamic method after more than one year keeping of the sample, dependencies of piezoelectric coefficient d_h on the pressure in the range of 0.1MPa to70MPa at different fixed temperatures from the interval 0^0C to 50^0C were investigated. It was determined linear increase of the value d_h with pressure and also increasing of the value d_h and $d(d_h)/dp$ coefficient with raise of the temperature. Equations of linear approximation of the experimental results $d_h(p)$ at temperatures 18^0 and 36^0C are following:

$$d_h[\mathrm{pC/N}]=39+0.016\cdot p[\mathrm{MPa}], \qquad (12)$$

$$d_h[\mathrm{pC/N}]=55+0.074\cdot p[\mathrm{MPa}]. \qquad (13)$$

Dependence ε'(T) doesn't depend on pressure and at temperatures 18^0 and 36^0C it equals to 36 and 56.

6. Conclusions

On the base of polycrystalline $Sn_2P_2S_6$ material and filling agent, using special treatment during polarization, we have received tablets of piezoelectric composite $Sn_2P_2S_6$, using special press-form. Physical parameters of the piezoelectric composite $Sn_2P_2S_6$ are following:

$$d_{33}\approx 12\ \mathrm{pC/N}; \quad \varepsilon'(f=1\mathrm{kHz})=38;$$

$$\varepsilon''(f=1\mathrm{kHz})=2; \quad d_h=119\ \mathrm{pC/N};$$

$$g_h=363\cdot 10^{-3}\ \mathrm{V\cdot m/N}.$$

From the received results it's seen, that piezoelectric tablets on the base of $Sn_2P_2S_6$ have competitive criteria. This is also confirmed by the results published in the article [15]. They are the suitable objects for investigation and working out of sensitive elements. For improvement of their quantitative indexes the finalizing of the preparation technology and the temperature treatment during polarization is required.

7. Acknowledgements

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ОДЕРЖАННЯ І ФІЗИЧНІ ВЛАСТИВОСТІ П'ЄЗОКОМПОЗИТІВ НА ОСНОВІ МАТЕРІАЛУ $Sn_2P_2S_6$

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Представлена технологія виготовлення п'єзоелектричних композитів $Sn_2P_2S_6$ у вигляді таблеток під дією тиску, температури та поляризуючого електричного поля. Досліджено частотні залежності імпедансу ($Z(f)$), фази ($\varphi(f)$), активного опору ($R(f)$), реактивного опору ($X(f)$), електричної ємності ($C(f)$), фактору втрат ($D(f)$), дійсної частини діелектричної проникливості ($\varepsilon'(f)$), уявної частини діелектричної проникливості ($\varepsilon''(f)$). Виявлено та визначено частоти «подавленого» резонансу – 151,0 кГц та 739,6 кГц. На частоті 1 кГц питомі параметри п'єзоелектричної таблетки $Sn_2P_2S_6$ є наступні: $Z^S(\mathrm{specific})\approx 1.92\cdot 10^6\,\Omega\cdot\mathrm{m}$, $\varphi\approx 1.513\,\mathrm{rad}$, $R^S(\mathrm{specific})\approx 2.91\cdot 10^7\,\Omega\cdot\mathrm{m}$, $X^S(\mathrm{specific})\approx 1.93\cdot 10^6\,\Omega\cdot\mathrm{m}$, $C\approx 2.1\cdot 10^{-11}\,\mathrm{F}$, $D\approx 0.057$, $\varepsilon'\approx 38$, $\varepsilon''\approx 2$. Визначені п'єзоелектричні коефіцієнти: $d_{33}\approx 12\,\mathrm{pC/N}$; $d_h\approx 119\,\mathrm{pC/N}$; $g_h\approx 363\cdot 10^{-3}\,\mathrm{V\cdot m/N}$. Динамічним методом було досліджено залежність d_h від тиску p в інтервалі тисків від 0.1 МПа до 70 МПа при різних фіксованих температурах з інтервалу від 0 °C до 50 °C. Спостерігався лінійний ріст величини d_h з тиском. Також величина d_h і коефіцієнт $d(d_h)/dp$ збільшувались з ростом температури.