

Acoustic and piezoelectric properties of new polar semiconductor compounds of $\text{Sn}_2\text{P}_2\text{S}_6$ family

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Introduction

The crystals of uniaxial ferroelectric $\text{Sn}_2\text{P}_2\text{S}_6$ family are very interesting for several reasons in the context of applications and basic research [1,2]. One reason is that these crystals exhibit strong piezoelectric effect and are photosensitive semiconductors. This offers a possibility for applications in acoustoelectronic and electroacoustic devices; consequently, investigations of acoustoelectric interaction are necessary. Another reason is that the sulphur substitution by selenium in a material leads to appearance of the incommensurate phase and the Lifshitz point providing unique possibility to investigate the critical behaviour of the phase diagram near such point [3,4]. Recently it was shown, that the mobile charge carriers also play essential role in the critical behaviour of crystals [5,6]. The variation of the electron density in energy levels can change the Lifshitz point position in the phase diagram and strongly modifies thermodynamic characteristics, such as optic and dielectric properties. Investigations of elastic and piezoelectric properties near Lifshitz point are also interesting. Recently the new layered crystals of this family were obtained: i.e. CuInP_2S_6 and CuCrP_2S_6 , which also are promising materials for microelectronics. In CuInP_2S_6 crystals the first order order-disorder phase transition occurs at $T_c = 312$ K, which is related to the hopping motions of Cu ions [7]. In the paraelectric phase crystal belongs to $C2/c$ symmetry group. Below T_c , in the ferroelectric phase with symmetry Cc spontaneous polarization appears and the polarization vector is oriented normally to the structure layers along c -axis. The spontaneous polarization is determined by two antiferroelectric contributions – ordering of Cu^+ ions and shifting of In^{3+} ions [8]. At $T < T_c$ the piezoeffect was found [9]. In layered CuCrP_2S_6 crystals, three consecutive phases were identified by means of differential scanning calorimetry and neutron powder diffraction: normal-nonpolar ($T > 190$ K), antipolar ($T < 150$ K) and intermediate phase ($150 < T < 190$ K) [8]. Diffraction investigations confirmed the following structures of room temperature and low temperature phases (monoclinic space groups $C2/c$ and Pc respectively). In the intermediate phase incomplete antipolar ordering of the copper was observed. This quasi-antipolar phase can be interpreted in terms of occasionally flipping dipoles in an otherwise antipolar phase, or static clusters of up and down dipoles that form “glassy” precursor to long range order. The nonpolar phase also was shown to be not of the usual paraelectric type, but, because of relatively strong dipole-dipole interactions, has polar clusters even at temperatures far above the transition. Here we present the investigation of phase transitions and ultrasonic, piezoelectric and

acoustoelectric properties of these $\text{Sn}_2\text{P}_2\text{S}_6$ family crystals. The ultrasonic and piezoelectric measurements were carried out by pulse-echo and resonance methods.

Results and discussion

The phase diagram of $\text{Sn}_2\text{P}_2(\text{Se}_x\text{S}_{1-x})_6$ crystals is complicated. Pure $\text{Sn}_2\text{P}_2\text{S}_6$ exhibits the second order ferroelectric phase transition (PT) near 337 K. At this PT the large critical slowing down in ultrasonic velocity is observed for all longitudinal ultrasonic modes. With substitution S by Se the PT temperature decreases and velocity anomalies increases (Fig. 1). In this figure the temperature dependencies of ultrasonic velocity measured at 10 MHz are shown for compounds with Se content $x=0$ (1), $x=0.15$ (2) and $x=0.28$ (3). The velocity anomalies at PT are accompanied by large and narrow ultrasonic attenuation peaks.

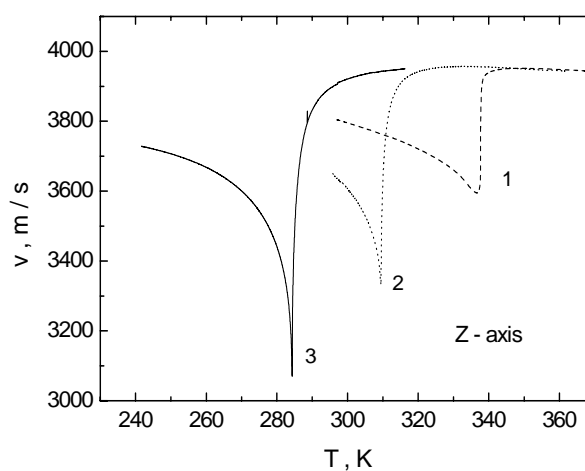


Fig.1. The temperature dependencies of longitudinal ultrasonic velocity in $\text{Sn}_2\text{P}_2(\text{Se}_x\text{S}_{1-x})_6$ single crystals for different Se content $x=0$ (1), $x=0.15$ (2) and $x=0.28$ (3)

It is interesting to note, that the critical exponent for anomalous part of ultrasonic velocity in paraelectric phase $\alpha = 0.5$ (Fig. 2), what is in good agreement with calculations according renormalization group theory [3]. For more detail discussion of anomalous ultrasonic attenuation, we refer to original papers [4,10].

With increase Se content above $x=0.28$ the PT splits to two and intermediate incommensurate (IC) phase appears. Therefore at $x_L=0.28$ and $T_L=283$ K the Lifshitz point exists in $\text{Sn}_2\text{P}_2(\text{S},\text{Se})_6$ system [2]. With further increase of x the temperature interval of existence of IC phase increases and for pure $\text{Sn}_2\text{P}_2\text{Se}_6$ it reaches $\Delta T_{IC}=30$ K

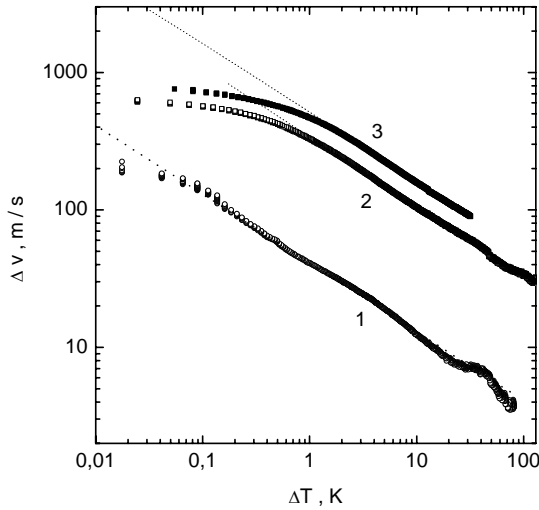


Fig.2. The temperature dependence of critical part of velocity in double logarithmic scale, in $\text{Sn}_2\text{P}_2(\text{Se}_x\text{S}_{1-x})_6$ single crystals for different Se content $x=0$ (1), $x=0.15$ (2) and $x=0.28$ (3)

(Fig. 3). The low temperature transition from IC phase to commensurate ferroelectric phase is the first order PT and the thermal hysteresis of about 1.5 K appears in heating and cooling measurement cycles.

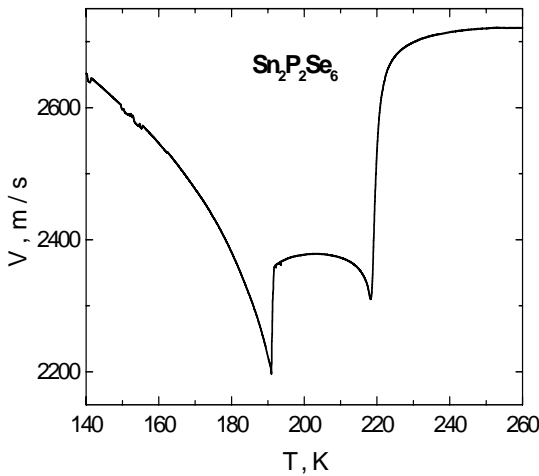


Fig.3. The temperature dependence of longitudinal ultrasonic velocity in $\text{Sn}_2\text{P}_2\text{Se}_6$ crystal along Y-axis

In the ferroelectric phase of $\text{Sn}_2\text{P}_2(\text{Se}_x\text{S}_{1-x})_6$ crystals the photosensitive ultrasonic attenuation caused by acoustoelectric interaction with free charge carriers was found for piezoactive modes. The dependencies of electronic attenuation coefficient α_{el} on electroconductivity σ of polarised crystals are presented in Fig. 4. Conductivity σ was changed by illumination of a sample. The photosensitivity of crystals is not very high and, at comparatively small σ , condition $\omega\tau \gg 1$ is fulfilled (ω -angular frequency of ultrasonic wave, τ -Maxwell relaxation time of electronic subsystem). In this case, α_{el} is independent on frequency and depends linearly on σ , what we observe in experiment. From this linear dependence, it

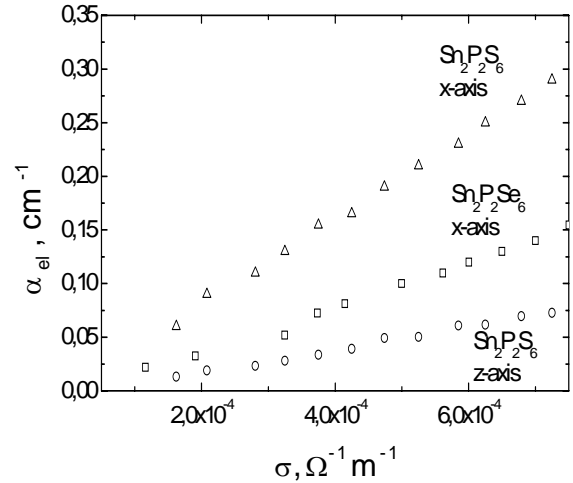


Fig.4. The dependence of α_{el} on conductivity σ of $\text{Sn}_2\text{P}_2\text{S}_6$ and $\text{Sn}_2\text{P}_2\text{Se}_6$ crystals in ferroelectric phase

was possible to calculate the values of electromechanical coupling parameters K (see [6] for details). The largest K values have been obtained for pure $\text{Sn}_2\text{P}_2\text{S}_6$ crystals: $K_{11}=0.7$, $K_{33}=0.17$. With increase of Se amount in the compound electro-mechanical coupling decreases and in pure $\text{Sn}_2\text{P}_2\text{Se}_6$ sample: $K_{11}=0.4$.

In CuInP_2S_6 single crystals the acoustoelectric interaction was weak, therefore we performed piezoelectric measurements using resonance method. We measured the

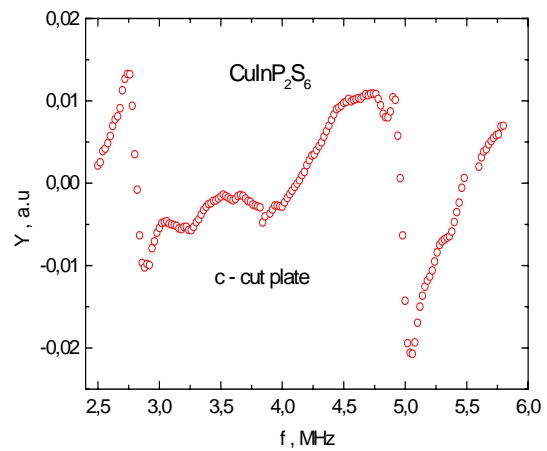


Fig.5. The frequency dependence of electric admittance of the CuInP_2S_6 crystal. The linear background is subtracted

frequency dependence of the modulus of electric admittance Y of a thin crystalline plate (thickness $d = 0.42$ mm). The orientation of the sample was such, that a.c electric field was directed along c - axis. The results are shown in Fig. 5. We attributed the antiresonance frequencies f_a to the thickness and shear vibrations of the CuInP_2S_6 plate. The antiresonance frequencies are observed when the thickness of sample matches the halfwavelength of shear or longitudinal elastic wave: $f_a = V/2d$. From this equation the values of ultrasonic velocities along c - direction were calculated at room temperature: $V_{SH} = 3320 \pm 50$ m/s; $V_L = 4460 \pm 50$ m/s. The square of the

electromechanical coupling coefficient was calculated for corresponding vibration modes from the equation:

$$K^2 = \frac{\pi f_r}{2f_a} \operatorname{tg}\left(\frac{\pi}{2} \frac{f_a - f_r}{f_a}\right). \quad (1)$$

At room temperature for longitudinal vibrations along ferroelectric c -axis the value $K_{33}^2 = 0.08$ for coefficient of electromechanical coupling was obtained. The K_{35}^2 value for shear mode is of the order of 0.05. These values are comparable to the electromechanical coupling coefficients of well known materials such as quartz or bismuth germanate.

The ultrasonic velocity and attenuation anomalies were recorded in layered CuInP_2S_6 samples at $T_c=310$ K (Fig. 6). Due to the small thickness of sample the data of ultrasonic attenuation are not very accurate. Dependence of ultrasonic velocity on temperature shows that there is a little softening at the phase transition temperature $T_c = 312.8$ K. In the

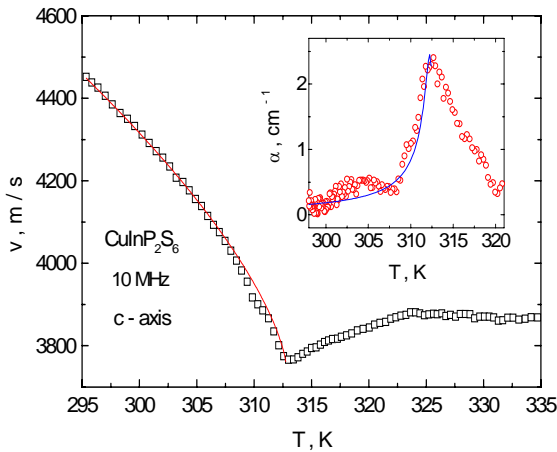


Fig.6. Dependence of ultrasonic velocity along the c -axis of the CuInP_2S_6 crystal on temperature. The solid line shows the least squares fit using Eq. (2). In the inset: ultrasonic attenuation vs. temperature (solid line - the approximation to Eq. 3)

ferroelectric phase additional contribution in ultrasonic velocity exists. Such behaviour is similar to that observed in DDSP and DMAAS ferroelectric crystals [10], where relaxation time of the order parameter (spontaneous polarisation) was comparatively long. In this case, the additional part of ultrasonic velocity in the ferroelectric phase was proportional to polarisation square (see [10] for details). Therefore it is possible to obtain critical parameter from this contribution. In order to obtain the critical exponent for temperature dependence of order parameter, bearing in mind that contribution of the quadratic term is prevailing in the low temperature phase, we approximated the ultrasonic velocity by equation:

$$v = v_0 + A(T - T_c)^{2\beta}, \quad (2)$$

where β is the critical exponent of the order parameter. The comparatively good least square fits (solid line in Figure 6) have been obtained using parameters; $T_c = 312.8 \pm 0.1$ K, $v_0 = 3755 \pm 5$ m/s, $A = 100 \pm 5$ and $\beta = 0.335 \pm 0.015$. The ultrasonic attenuation in low temperature phase arises from

the elastic strain - polarisation interaction and can be described by the Landau Chalaitnikov theory [11]:

$$\alpha = 2\alpha_{\max} \frac{\omega\tau}{1 + \omega^2\tau^2}, \quad (3)$$

here α_{\max} - the attenuation coefficient at maximum, ω - the angular frequency of elastic wave and $\tau = \tau_0 / (T_c - T)$ - the polarisation relaxation time, which increases critically approaching T_c . The solid line is drawn in the inset of Fig. 6 by approximation according to Eq. 3 ($\tau_0 = 7.2 \times 10^{-9}$ s×K). The long polarisation relaxation time was also obtained from dielectric experiments, where the low frequency dielectric dispersion was observed [12]. Above T_c the pretransitional effects in ultrasonic and piezoelectric properties are clearly seen. Such effects can be determined by polar clusters existing in paraelectric phase.

Further, in this contribution, we present similar as in CuInP_2S_6 measurements of layered CuCrP_2S_6 . As it was described in introduction, two successive phase transitions are expected to occur in these crystals. It was confirmed by our ultrasonic velocity and attenuation measurements. The temperature dependencies of longitudinal ultrasonic velocity and attenuation have been measured along c -axis at 10 MHz frequencies in CuCrP_2S_6 crystals and anomalies at the phase transition have been observed (Fig.7 and 8). Due to the small thickness of sample ($d=0.14$ mm) the data of ultrasonic attenuation and velocity are not very perfect. The temperature dependence of ultrasonic velocity shows that there are two minima near temperatures $T_1=180$ K and $T_2=145$ K (Fig.7). Velocity minima correspond to the attenuation peaks (Fig.8). Such ultrasonic behaviour is in very good agreement with colorimetric data [13]. The attenuation has additional contribution, which increases with temperature increasing. It can be determined by the influence of high ionic conductivity of CuCrP_2S_6 crystals as in other fast ionic conductors [14]. Indeed, the ionic conductivity, according to [15], is comparatively high for CuCrP_2S_6 and CuInP_2S_6 crystals. The long tail in ultrasonic velocity in the high temperature phase ($T>180$ K) of CuCrP_2S_6 as in case of CuInP_2S_6 may be caused by polar clusters, which were mentioned in introduction. The critical

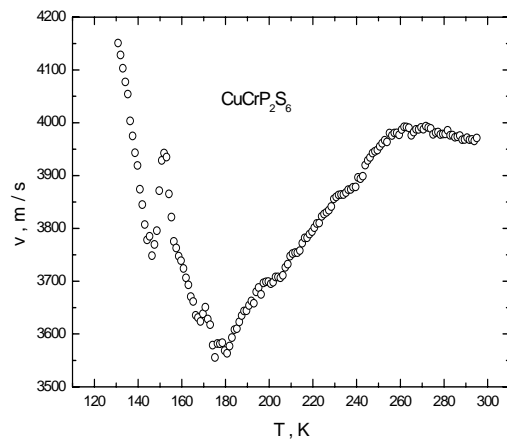


Fig. 7. The temperature dependence of longitudinal ultrasonic velocity measured along c -axis of the CuCrP_2S_6 crystal

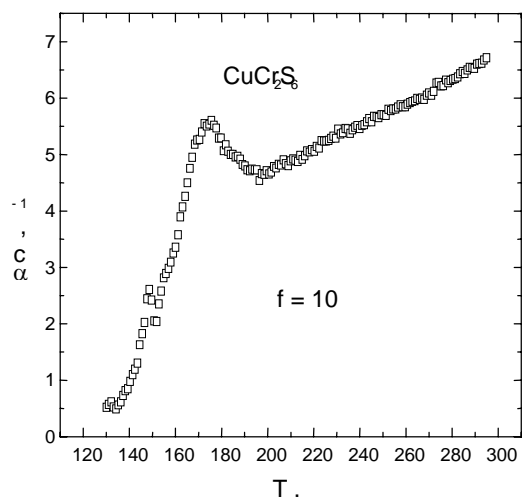


Fig.8. The temperature dependence of ultrasonic attenuation measured along c-axis of CuCrP_2S_6 sample

contribution of defects is also possible in high temperature phase. The steep increase of velocity in antipolar phase (below 145 K) is similar to that which was observed in DDSP, DMAAS and CuInP_2S_6 single crystals [9,10,16], where the relaxation time of the order parameter was comparatively long. In this case the temperature dependence of ultrasonic velocity in the low temperature phase was proportional to square of order parameter. In order to confirm the existence of piezoeffect we measured the frequency dependence of modulus of electric admittance Y for thin crystalline plate (thickness $d = 0.14$ mm). The orientation of the sample was such, that a.c electric field was directed along c - axis of the CuCrP_2S_6 crystal. The results are shown in Fig. 9. The resonance and antiresonance character in admittance Y frequency dependence is clearly seen near 15 MHz frequency. We attributed the resonance frequencies f_a to the thickness vibrations of the CuCrP_2S_6 plate. The antiresonance frequency is observed when the thickness of sample

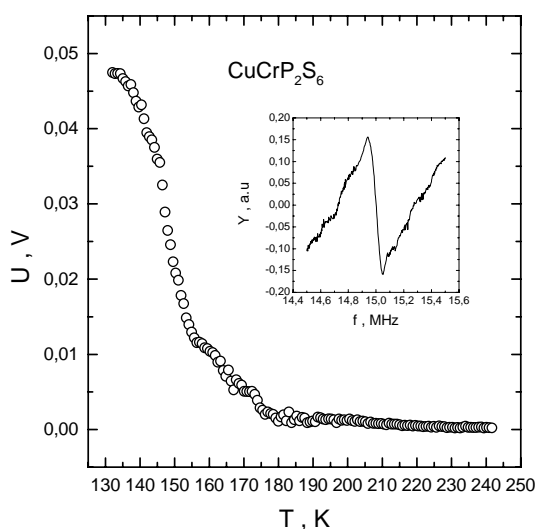


Fig.9. The temperature dependence of electric signal detected by CuCrP_2S_6 plate. In the inset: the frequency dependence of electric admittance of the CuCrP_2S_6 plate at 130 K temperature. The linear background is subtracted

matches the half wavelength of longitudinal elastic wave: $f_a = V/2d$. From the equation the values of ultrasonic velocity along c - direction have been calculated at temperature $T=130$ K: $V_L = 4160 \pm 50$ m/s. This value we used for calibration of ultrasonic velocity measured by pulse echo method (Fig.7). The square of the electromechanical coupling coefficient was calculated for this vibration mode from the equation (1). The K_{33}^2 value of 2 % has been obtained for the longitudinal mode of CuCrP_2S_6 crystal. This value is less than corresponding value for CuInP_2S_6 crystals. In order to confirm the existence of the piezoeffect in CuCrP_2S_6 we made direct experiment on the same thin 0.14 mm plate working as a piezoelectric transducer. The longitudinal ultrasonic wave was excited by LiNbO_3 transducer on 10 MHz frequency and was detected by CuCrP_2S_6 plate at $T=130$ K temperature. The efficiency of such transducer was quite good. The temperature dependence of detected by CuCrP_2S_6 transducer signal was measured (Fig.9) and it was shown, that this signal drops down at $T=145$ K and completely disappears in the paraelectric phase (above $T = 190$ K). If we assume that the amplitude of detected by ultrasonic transducer signal is roughly proportional to the square of electromechanical coupling coefficient, which somewhat shows the polarisation of material, we can conclude that, below $T=145$ K the antipolar phase exists and in intermediate phase ($145 < T < 180$ K) the quasiantipolar phase is present in CuCrP_2S_6 crystal.

The other photosensitive ultrasonic phenomena such as acoustoelectric voltage, temperature oscillations, relaxing intermediate-incommensurate phase and photostimulated shift of PT temperature were observed and investigated in all these polar crystals of $\text{Sn}_2\text{P}_2\text{S}_6$ family. Illumination induces the intermediate-IC phase not only in the compound $\text{Sn}_2\text{P}_2(\text{Se}_{0.28}\text{S}_{0.72})_6$ where the Lifshitz point exists, but also in pure $\text{Sn}_2\text{P}_2\text{S}_6$ crystal [6]. For investigation of photo stimulation phenomena in $\text{Sn}_2\text{P}_2\text{S}_6$ crystals at first the sample was annealed in dark at 320 K temperature and we took measurements of ultrasonic velocity $v = f(T)$ and attenuation $\alpha = f(T)$ in cooling run. The results for longitudinal wave along Y - axis are shown in Fig. 10 (lines 1). The temperature dependencies of $v = f(T)$ and $\alpha = f(T)$ are completely the same as we observed previously [17]. After that, the sample was heated up to 320 K and measurements of velocity and attenuation were resumed in illuminated sample. In later case the shift of $v = f(T)$ and $\alpha = f(T)$ anomalies to lower temperature was observed (Fig.7, lines 2). The shift of incommensurate PT: $\Delta T_i \approx 1.6$ K, and that of the ferroelectric PT: $\Delta T_C \approx 2.1$ K. It is interesting to note that the dependence of velocity is modified in all ferroelectric phase. Such behaviour we attribute to the photodomain effect: i.e. free charge carriers are screening the polarisation and internal electric field changes under illumination. Also the influence of acoustoelectric field can not be ruled out. The phenomenological theory of photostimulated shift of the ferroelectric PT is developed in [18]. There it was shown that: $\Delta T_C = -(C/2\pi)aN$, where C -Curie constant, a - coefficient of electron phonon interaction, N - the total density of charge carriers. Our experiment does not contradict this theory. The relaxation time of

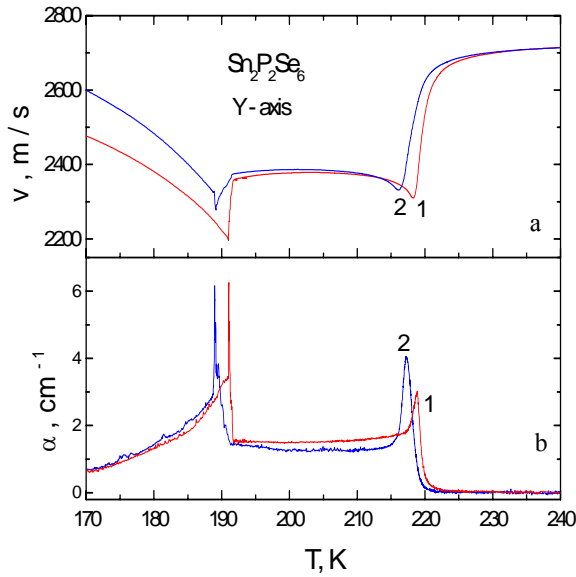


Fig.10. The temperature dependencies of ultrasonic velocity (a) and attenuation coefficient measured along Y-axis in $\text{Sn}_2\text{P}_2\text{Se}_6$ crystal. Sample in dark (1), illuminated sample (2)

photostimulated effect we measured using chopped light beam and it was longer than 30 s for $\text{Sn}_2\text{P}_2\text{Se}_6$ samples. It shows that the main reason of observed photostimulated phenomena is the redistribution of charge carriers in deep energy levels.

Another interesting phenomenon was the ultrasonic velocity and attenuation oscillations, which were observed in incommensurate phase of $\text{Sn}_2\text{P}_2\text{Se}_6$ sample in conditions of very slow heating. The temperature dependencies of these oscillations are shown in Fig.11. The correspondence between velocity and attenuation behaviour is clearly seen. Similar oscillations were observed before in optical

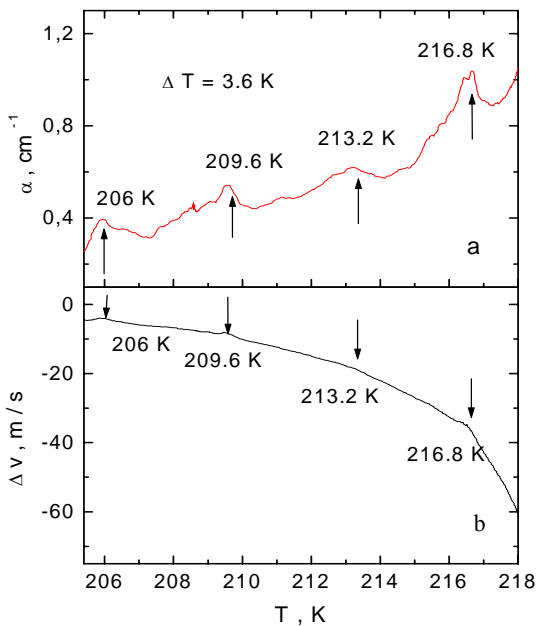


Fig.11. The ultrasonic attenuation (a) and velocity (b) oscillations in the incommensurate phase of $\text{Sn}_2\text{P}_2\text{Se}_6$ crystal

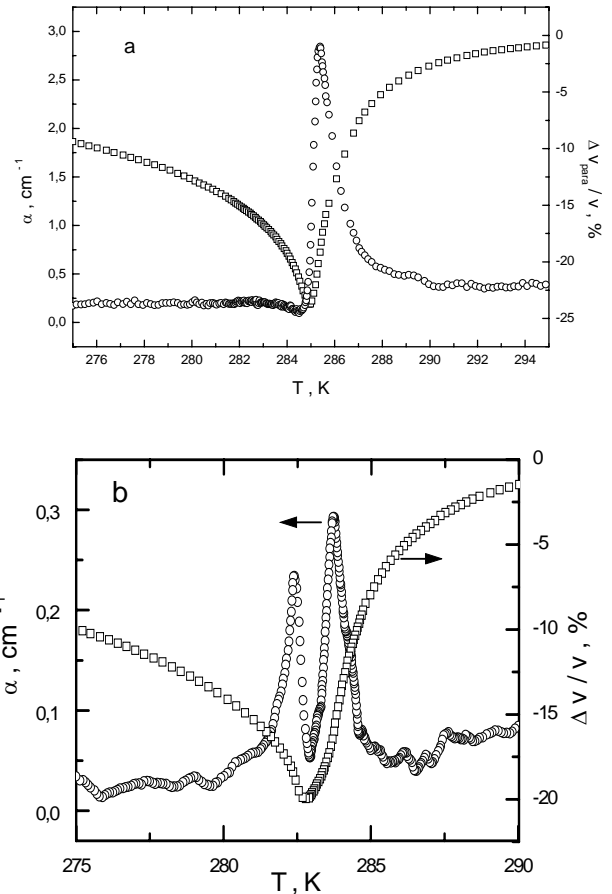


Fig.12. The dependence of ultrasonic velocity and attenuation on temperature measured along Z-axis of $\text{Sn}_2\text{P}_2\text{S}_{0.28}\text{Se}_{0.72}$ crystal. Sample in dark (a), illuminated sample (b)

experiments in $\text{Sn}_2\text{P}_2\text{Se}_6$ crystal and in dielectric measurements of the IC phase of urea doped thiourea [19,20]. To our mind this phenomenon is closely related to the nature of IC phase.

Special attention was paid to the $\text{Sn}_2\text{P}_2\text{S}_{0.28}\text{Se}_{0.72}$ compound where the Lifshitz point exists. Original ultrasonic studies of this material revealed ultrasonic anomalies as large as to make us to choose the Z-axis along which elastic anomalies were the smallest. The $\Delta v/v = f(T)$ and $\alpha = f(T)$ curves are shown in Figure 12a. The attenuation anomaly is wider in the paraelectric phase showing a strong influence of fluctuations which increase at the Lifshitz point. The most interesting result was obtained when measurement was performed on illuminated sample: apart from the shift of phase transition to lower temperature splitting of the transition was observed (Figure 12b). Two sharp attenuation peaks are clearly seen indicating that illumination at the Lifshitz point leads to appearance of the incommensurate phase. The width of attenuation peaks shows that the order parameter relaxation time decreases when the charge carrier density is increased by illumination. The photosensitive ultrasonic phenomena in $\text{Sn}_2\text{P}_2\text{S}_{0.28}\text{Se}_{0.72}$ caused by the shift of the transition exist far in the paraelectric phase >50 K above

T_C , where we were able to detect small ultrasonic velocity variations.

Conclusions

To summarise: all investigated materials have strong piezoelectric effect, nevertheless they are wide band gap semiconductors and charge carriers in deep energy levels create space charge, which modifies the elastic, dielectric and, as a result, electroacoustic parameters. This manifests itself in long time relaxation of these properties, especially near PT and in IC phase. So, for applications it is important to verify the conditions for the optimisation of electroacoustic parameters.

Acknowledgement

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V. Samulionis

Naujų polinių puslaidininkinių $\text{Sn}_2\text{P}_2\text{S}_6$ šeimos junginių ultragarsinės ir pjezoelektrinės savybės

Reziumė

Straipsnyje pateikiama $\text{Sn}_2\text{P}_2\text{S}_6$ šeimos junginių ultragarsinių ir pjezoelektrinių savybių tyrimo apžvalga. Šios medžiagos yra feroelektrikai - puslaidininkiai, kuriuose žemos temperatūros fazėje susidaro gana stiprus pjezoeftektas. Fazinių virsmų aplinkoje šiuose kristaluose atsiranda anomalūs ultragarso slopinimas ir greičio dispersija. Pastebėta nemaža fotojautrio reiškinų: akustoelektrinė sąveika, ultragarso greičio ir slopinimo osciliacijos nebendramatėje fazėje, fotostimuliuotas fazinio virsmo poslinkis žemų temperatūrų link ir fazinio virsmo skilimas į du Lifšico taško aplinkoje. Manoma, kad visi šie reiškiniai atsiranda dėl krūvininkų persiskirstymo ir relaksacijos giliuose energijos lygmenyse.

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