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EXPERIMENTAL STUDY OF ELECTRIC FIELD INFLUENCE ON LOW-TEMPERATURE LONG-TIME RELAXATION IN CRYSTALLINE FERROELECTRICS

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1. Introduction

Low-temperature thermal properties (heat capacity, heat conductivity) of crystalline ferroelectrics (single crystals or polycrystalline) with a diffuse phase transition (PLZT, SEN, PMN, PMT, PSN) resemble amorphous solids 1 - 11: they show excess heat capacity, roughly proportional to T, T^2 dependence of the heat conductivity below ~1 K, and a plateau near 10 K on the heat-conductivity temperature dependence. On the contrary, such anomalies for ferroelectrics with a sharp phase transition are at least essentially smaller and possibly due to randomly distributed dipolar defects 11 - 13. Thus, no heat conductivity plateau was detected in most of these ferroelectrics 11 - 13.

According to recent experiments 14 - 21, long-time energy relaxation also seems to be a common feature of amorphous solids: power release $Q(T_1, T_0, t)$ observed after rapid cooling , from the initial temperature T_1 to the final temperature T_0 relaxes very slowly, roughly proportional to t_7^{-1} .

To answer the question about such behaviour of crystalline ferroelectrics, we have measured the heat capacity and power release of two various specimens: polycrystalline $Pb_{0.915}La_{0.085}$. $(2r_{0.65}Ti_{0.35})O_3$ (PLZT, mass m = 50.6 g) with a diffuse phase transition at $T_c = 370$ K, and a sharp-phase-transition single crystal of KH_2PO_4 (KDP, m = 94.8 g). At the same time, we have studied the influence of the static electric field on these phenomena in PLZT. Some of the preliminary results already appeared in Ref. 22.

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2. Model

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At temperatures low enough $(T_0, T_1 \lesssim 2 \text{ K})$, long-time relaxation observed in amorphous solids can be treated within the standard phenomenological tunneling model ²³, ²⁴. If one assumes uniform distribution of two-level systems,

$$P(\Delta, \lambda) = P = const. , \qquad (1)$$

where Δ is the asymmetry energy and λ is the tunneling parameter, the standard tunneling theory gives a time dependent density of states

$$n(\epsilon, t) = n_0(t) = (\bar{P}/2) \ln(4t/\tau_{min})$$
, (2)

a time dependent heat capacity

$$c_t = (\pi^2 k_B^2 / 12) \bar{P} Tln(4t / \tau_{min})$$
, (3)

and a corresponding power release (absorption) after cooling (heating) the system from the equilibrium temperature T_1 to the temperature T_0

$$\dot{Q}(T_1, T_0, t) = V(\pi^2 k_B^2/24) \bar{P}(T_1^2 - T_0^2) t^{-1}$$
, (4)

where ϵ is the energy gap between the two levels, τ_{\min} is the shortest relaxation time and V is the volume of the specimen.

For higher temperatures, better agreement with experimental data is obtained using the energy dependent distribution function $19,\ 20$

$$n(\epsilon, t) = n_0(t)/(1 + exp((\epsilon - \epsilon_1)/(k_B T_b)))$$
, (5)

where $\epsilon_f = k_B T_f$ and T_b are constant. With (4), equations (3) and (4) are transformed into

$$\mathbf{c_{t}} = (\pi^{2} \mathbf{k}_{B}^{2} / 12) \overline{P} \mathbf{I}_{1} (\mathbf{T} / \mathbf{T}_{f}, \mathbf{T}_{b}) \mathbf{T} \ln(4t / \tau_{min})$$
(6)

and

$$\dot{Q}(T_1, T_0, t) = V(\pi^2 k_B^2/24) \overline{P}(I_2(T_1/T_f, T_b)T_1^2 - I_2(T_0/T_f, T_b)T_0^2)t^{-1}$$
(7)

where

$$I_{1}(T/T_{f},T_{b})=(6/\pi^{2})\int_{0}^{\infty} \frac{x^{2}\exp(-x)dx}{(1+\exp(-x))^{2}(1+\exp((xT/T_{f}-1)/(T_{b}/T_{f})))},$$

$$I_{2}(T/T_{f},T_{b})=2(T_{f}/T)^{2}\int_{0}^{T/T_{f}} (T/T_{f})I_{1}(T/T_{f},T_{b})d(T/T_{f}).$$
(9)

Moreover, for large t it is necessary to take into account the maximum tunneling parameter $\lambda = \lambda_{\max}$ (and corresponding $\tau = \tau_{\max}$). If we assume, instead of (1),

$$P(\Delta, \lambda) = \overline{P} \quad \text{for} \quad \tau \leq \tau_{\max},$$

$$P(\Delta, \lambda) = 0 \quad \text{for} \quad \tau > \tau_{\max}, \quad (10)$$
we get, for $t >> \tau_{\min}, 20$

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$$\dot{q}(\mathbf{T}_{1},\mathbf{T}_{0},t) = V\overline{P}(\pi^{2}k_{B}^{2}/24)(\mathbf{I}_{2}(\mathbf{T}_{1}/\mathbf{T}_{f},\mathbf{T}_{b})\mathbf{T}_{1}^{2}-\mathbf{I}_{2}(\mathbf{T}_{0}/\mathbf{T}_{f},\mathbf{T}_{b})\mathbf{T}_{0}^{2})t^{-1}exp(-t/\tau_{max})$$
(11)

instead of (7): the usual t^{-1} dependence is transformed into an exponential one for time $t > \tau_{max}$. Up to now, there has been no experimental evidence of such behaviour in amorphous solids - real experiments lasted probably too little compared to τ_{max} .

3. Experimental

First, low-temperature heat capacity and heat release were measured in the absence of electric field.

The experimental arrangement was the same as in Ref. 26. The specimen with the SI germanium thermometric resistor, a heater and the contact for the mechanical heat switch (about 5.5 g of copper, less than 10 mg of glue) hangs in the calorimeter on 4 kapron threads (0.13 mm in diameter, 30 mm long).

The heat capacity measurements were quite standard. During the heating of the specimen, the temperature of the body of the calorimeter T_K was regulated to be approximately the same as the rising temperature T of the specimen.

For heat release study, the resistance drift R of the thermometer was measured as a function of time t after cooling the specimen from the equilibrium temperature T_1 (where the specimen remained at least for 10 hours) to T_0 . The corresponding power released was then determined from the directly measured power release $\dot{Q}_{_{\rm I\!I\!I}}$, the heat leak $\dot{Q}_{_{\rm I\!I}}$ and the heat background $\dot{Q}_{_{\rm I\!I\!I}}$ as

$$\dot{Q}(T_1, T_0, t) = \dot{Q}_m - \dot{Q}_A - \dot{Q}_B$$
, (12)

where

$$\dot{q}_{m} = \dot{R}C/(\partial R/\partial T)$$
, (13)

$$\dot{Q}_{A} = A(T_{K} - T)$$
 (14)

C is the heat capacity of the specimen, $\partial R / \partial T$ is the sensitivity of the thermometer, and A is the coupling constant between the specimen and the body of the calorimeter. Usually, $T_{\rm K} \simeq T$. The parameters A and $\dot{Q}_{\rm B}$ were determined experimentally.

The accuracy of the measurement is limited by the time fluctuations of $\dot{Q}_{\rm R}$, approximately equal to 100 pW.

Since neither significant heat-capacity anomalies nor heat release were observed in KDP, the electrical field influence was studied for PLZT only. Thin layers of aluminium paste (their total mass was 0.16 g), deposited on the upper and lower bases of the specimen, form the electrodes. Before the experiment, the specimen was kept in static electric field (4.0 kV/cm) at 200° C ($T_{c} \simeq 100^{\circ}$ C) for two hours and cooled down slowly (in the field) to the room temperature. Then, after switching off the field, the copper foil with the thermometer, heater and contact to the heat switch was glued to the specimen. After cooling to helium temperatures, the

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heat capacity and the power release were measured for various electric field strengths (E = 0, 1.7 kV/cm, 2.2 kV/cm, 2.6 kV/cm, 3.5 kV/cm, 4.3 kV/cm) again. The power released after switching on and off the electric field was also studied at various temperatures.

4. Results

4.1 Heat capacity

The results of our heat capacity measurements are shown in fig. 1. In agreement with previous experiments 5, PLZT heat capacity contains a rather large term roughly proportional to the temperature at T < 3 K. This anomaly appeared to be a little smaller after the heat treatment of the specimen in electric field, and is much smaller in KDP. Assuming

$$c = aT + bT^3$$
, (15)

we get a = $(3.1 \pm 0.2) \ \mu J/gK^2$, b = $(0.8 \pm 0.2) \ \mu J/gK^4$ for PLZT before the treatment, a = $(2.5 \pm 0.2) \ \mu J/gK^2$, b = $(0.8 \pm 0.2) \ \mu J/gK^4$ for PLZT after the treatment, and a = $(0.5 \pm 0.1) \ \mu J/gK^2$, b = $(2.05 \pm 0.10) \ \mu J/gK^4$ for KDP.

Heat capacity measurements in the static electric field ($E \le 4.3 \text{ kV/cm}$) showed no measurable influence of the field on the heat capacity data.

4.2 Heat release

No heat release was observed in the KDP single crystal; more accurately, the power measured 10 minutes after the beginning of the cooling from $T_1 = 10$ K to $T_0 = 1.3$ K equaled the heat

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background within the accuracy of our measurements. (The time of the cooling was about 5 minutes.)

The specific heat release $\dot{q} = \dot{Q}/m$ in PLZT before the treatment for various initial temperatures T_1 , and $T_0 = 1.3$ K is shown in fig. 2. The procedure was as follows: after the relaxation was completed at 1.3 K, the specimen was successively heated to various T_1 and kept at this temperature for $t_H = 10$ h (open dots) or $t_H = 5$ min (full dots); after another cooling

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to 1.3 K, the time dependence of $q(T_1, 1.3 \text{ K}, t)$ was measured again.

For low T_1 , heat release \dot{q} increases with T_1 . Above 13 K, \dot{q} is T_1 independent. Whereas \dot{q} is proportional to t^{-1} for not too large t, it decreases exponentially in time for large t at variance with vitreous silica 17 and amorphous metals $^{18} - ^{20}$. Experimental points lie on the curves

$$\dot{q} = q_0 t^{-1} \exp(-t/\tau_{max}) , \qquad (16)$$

surprisingly well corresponding to (11), if $\bar{P}_{m} = \bar{P}/\beta = 1.45^{\circ}10^{38}/Jg$, where β is the density of the specimen, (smaller value \bar{P}_{m} of Ref. 22 is the result of fitting with equation (7) without taking into account $\exp(-10 \text{ min}/\tau_{max}) \neq 1$), $T_{f} = 7.5 \text{ K}$, $T_{b} = 0$, and values of T_{1} -dependent τ_{max} according to the inset in fig. 2. The heat release after the treatment is smaller for the same T_{1} , T_{0} for not too large t (fig. 3), yielding $\bar{P}_{m} = 1.2^{\circ}10^{38}/Jg$, and T_{f} , T_{b} , τ_{max} being unchanged.

We have not observed any influence of the electric field on relaxation, when both the cooling from T_1 to T_0 and the



Fig. 3. q_0 for various T_1 ($T_0 = 1.3$ K)

O - q_0 before the heat treatment, • - q_0 after the heat treatment, 1 - (11) with $\bar{P}_m = 1.45^{\circ}10^{38}/J_g$, $T_f = 7.5$ K, $T_b = 0$, 2 - (11) with $\bar{P}_m = 1.20^{\circ}10^{38}/J_g$, $T_f = 7.5$ K, $T_b = 0$, 3 - (4) with $\bar{P}_m = 1.45^{\circ}10^{38}/J_g$.

heat release measurements were performed in the field. (This corresponds to the field independence of the heat capacity).

On the other hand, a long-time heat release was observed after switching on or off the field at a definite temperature (fig. 4). The <u>release</u> is observed in both cases (switching on and off), and its values are the same for the same absolute value of field change (except the release after the first field change) with a good reproducibility of the results. For example, curve 1 in fig. 4 (heat release after changing the field from 0 to 1.72 kV/cm and vice versa) was measured ten times and all experimental points agree within 10 per cent. Only the heat release after the first field change (either switching on or switching off) after the cooling was about 50 per cent larger.



Fig. 4. Relaxation of the heat release after switching on or

off the electric field at 1.3 K

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O - E = 1.7 kV/cm, ● - E = 2.2 kV/cm, △ - E = 2.6 kV/ ▲ - E = 3.5 kV/cm, □ - E = 4.3 kV/cm Full curves: (16) with $q_0(E)$ and $\tau_{max}(E)$ according to figs. 6,7; 1 - E = 1.7 kV/cm, 2 - E = 2.2 kV/cm, 3 - E = 2.6 kV/cm, 4 - E = 3.5 kV/cm, 5 - E = 4.3 kV/cm.

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No variations of field induced long-time relaxation were observed, when the measuring temperature was changed from 1.15 K to 3 K.

The total heat released during the first two minutes after an electric field change $0 \rightarrow E$ or $E \rightarrow 0$ is shown in fig. 5. Since this heat is partially due to the usual dielectric losses, it is essentially larger when the field is switched off because of smaller time constant than at switching it on.

Any influence of the electric field on the background power was not seen.

The time dependence of the field-induced long-time power release is similar to that induced by changing the temperature (fig. 2): time dependence

$$\dot{q}(E,t) = q_0(E)t^{-1}exp(-t/\tau_{max})$$
, (17)



Fig. 5. Total heat released during the first two minutes after

the electric field switching

O - after switching off, **④** - after switching on;
1 -
$$\triangle Q = 0.86E^2$$
, 2 - $\triangle Q = 1.31E^2$, $[\triangle Q] = \mu J$,
[E] = kV/cm.

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analogous to (11), gives a good fit to the experimental data (fig. 4) with τ_{max} proportional to E (fig. 6) and $q_0(E)$ proportional to E^2 (fig. 7).

5. Discussion

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The FLZT heat capacity contains a rather large term roughly proportional to T, of the same order of magnitude as in vitreous silica (a $\approx 1.2 \ \mu J/gK^2$). Since the dependence of the parameter \bar{P}_m on τ_{min} is not strong, we have tried to *estimate \bar{P}_m from (6) using vitreous silica $\tau_{min} = 4.5 \ x$ $10^{-10} T^{-3} sec$, $[T] = K^{26}$. We have obtained $\bar{P}_m = 7.2^{\circ} 10^{38}/Jg$.

It is difficult to choose between (3) and (6), when T_{f} is large. With $T_{f} = 7.5$ K for PL2T, more detailed analysis ²² shows fit with (6) is really better than with (3).

For t << τ_{max} , the power release is proportional to t⁻¹. Using T₁ dependence of the power release for small T₁, we get $\overline{P}_{m} = 1.45^{\circ} 10^{38}/J_{g}$, five times smaller value than that from heat capacity measurements. This resembles vitreous silica data, where heat-capacity \bar{P}_m is about twice as large as the power-release \bar{P}_m .

Equation (7), resp. (11) agree with the experimental data well even for higher T_1 (figs. 2,3). The corresponding $T_f =$ 7.5 K is smaller than that for vitreous silica (13 K) and for amorphous $Fe_{80}B_{14}Si_6$ (20 K) or $Co_{69}Fe_{4.5}Cr_2Si_{2.5}B_{22}$ (24 K).

Similarly to amorphous metals ^{19, 20} and epoxy resin ²¹, relaxation at $T_o > T_f / 10$ is faster than according to the standard tunneling theory. For instance, 1.3 K heat release after $t_H = 5$ min at $T_H = 4.5$ K is almost the same as after $t_H = 10$ hours at the same T_H (see fig. 2). According to the phenomenological expression for the ratio of power release after a short heating time t_H at T_H to that after a very long ($t_H \rightarrow \infty$, $T_H = T_1$) heating time, developed for amorphous metals ^{19, 20},

$$\mathbf{r} = \dot{q}(\mathbf{T}_{H}, \mathbf{T}_{O}, \mathbf{t}_{H}, \mathbf{t}) / \dot{q}(\mathbf{T}_{1}, \mathbf{T}_{O}, \mathbf{t}) = (1 + (t/t_{H})f(\mathbf{T}_{H}) / f(\mathbf{T}_{O}))^{-1},$$

where

$$f(T) = \exp(-6T/T_{e}),$$

we get r = 0.87 for PLZT ($T_f = 7.5$ K) when T = 4.5 K, $T_o = 1.3$ K, $t_H = 5$ min., t = 10 min. This value is very close to the experimental $r = 0.86 \pm 0.05$.

Taking all these facts into account, it seems reasonable to conclude that the low-temperature behaviour of PLZT is a typical behaviour of an amorphous structure.

On the other hand, such glassy properties are absent in KDP: no long-time relaxation is observed, and the T term of the heat capacity is much smaller. Such result corresponds well

to the conclusion of Gmelin et al. ^{12, 13} and Yoreo et al. ¹⁰ that the glassy-like behaviour is really observed for ferroelectrics with diffuse phase transition only.

PLZT significantly differs from amorphous solids in the possibility - up to now unique - of τ_{max} determination, studying the heat release at t \simeq au_{max} . According to the experimental data, shown in the inset in fig. 2, $\tau_{\rm mer}$ is proportional to T_1 for $T_1 < T_f$, and is independent of T_1 , if $T_1 > T_f$: the level systems with maximum relaxation time are those with maximum energy $\epsilon_1 = k_B T_1$. According to distribution (5), systems with $\epsilon_1 > \epsilon_r$ are absent and τ_{max} , corresponding to $\epsilon_r = k_B T_r$, is T_1 independent for $T_1 > T_r$. Experimental τ_{max} are at least 100 times larger in vitreous silica ¹⁷, amorphous metals 18 - 20 or epoxy resin ²¹ than for PLZT. It would be of interest to check this feature in other ferroelectrics with the diffuse phase transition (SBN, PSN, PZT, PMN). The ferroelectric (KBr)_{1-x}(KCN)_x is an interesting crystalline system, too. For $0.25 \le x \le 0.7$, a glassy behaviour of the low-temperature heat conductivity and heat capacity including its time dependence has been observed in good agreement with (6) for 10^{-4} sec $\leq t \leq 10$ sec $\frac{10}{10}$.

The treatment with cooling the specimen in static electric field through the temperature T_c of the phase transition results in a small effect only: both the heat capacity linear term and the heat release at time not too large are about 20 per cent smaller. So the electric field (and/or possibly the tempering the specimen) influences equally two-level systems with both short and long relaxation time. However, the changes are not large enough to allow systematic study of the field influence on the phase transition, our field E = 4 kV/cm is probably too small to influence the polarization structure significantly.

No influence of the electric field on the characteristics of the two-level systems has been found: neither the heat capacity, nor the power release depends on the external electric field at least for $E \leq 4.3$ kV/cm.

On the other hand, long-time relaxation observed after switching on or off the field indicates that the electric field changes involve changes in the charge distribution via ion tunneling. At the same time, only tunneling with large relaxation time takes part in this heat release: neglecting usual dielectric losses, we obtain a lower limit for the effective τ_{\min} from the measurement of the total heat AQ(2 min.) released in the time interval $\tau_{\min} \leq t \leq 2 \min$, if extrapolating t⁻¹ dependence of power release to τ_{\min} :

 $\Delta Q(2 \text{ min.}) \int_{\mathcal{T}_{min}} q(E)t^{-1}dt = q(E)\ln(2 \text{ min.}/\tau_{min})$ (18)

$$\tau_{\min} > 2 \min \cdot \exp(-\Delta Q/q_0) \simeq 1 \sec$$
 (19)

according to figs. 5 and 7.

Nevertheless, the same time dependence of the heat release after rapid cooling and after electric field change for large t indicate that the mechanisms of both relaxation processes are similar. However, a more detailed theoretical analysis is necessary for better understanding.

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6. Conclusions

1. Long-time low-temperature heat relaxation typical of amorphous solids has been observed in the polycrystalline ferroelectric PLZT.

For sufficiently low temperatures $T_1 < 2 \text{ K}$ and $T_0 = 1.3 \text{ K}$, experimental heat release agrees with the standard tunneling theory well. It enables one to determine the distribution parameter of two-level systems $\overline{P}_m = 1.45 \cdot 10^{38}/\text{Jg}$, which is about five times smaller than the value calculated from heat capacity measurements.

 T_1 dependence of power release for $T_1 > 2 \text{ K}$ ($T_0 = 1.3 \text{ K}$) can be understood within the modified version of the tunneling model, assuming the existence of a maximum energy $\epsilon_f/k_B =$ 7.5 K in the distribution function of the density of states, which is not far from the corresponding ϵ_f values of various amorphous solids (13 K $\leq T_f \leq 24 \text{ K}$).

For $T_o > 1.3$ K, the relaxation is faster than according to the standard model. A similar behaviour was found for glasses, too.

• For large measuring time, the typical amorphous t^{-1} dependence is transformed into an exponential one. The maximum relaxation time τ_{max} is proportional to T_1 for $T_1 < 7.5$ K and independent of T_1 for $T_1 > 7.5$ K. The maximum relaxation time $\tau_{max} \simeq 3$ hours ($T_1 = T_f$, $T_o = 1.3$ K) is essentially smaller than in amorphous solids.

2. The excess heat capacity of KH_2PO_4 is much smaller than that of PLZT, and long-time heat relaxation was not observed. These results agree with the hypothesis that only those pure ferroelectrics whose phase transition is diffuse show a glassy behaviour.

3. The power release and the excess heat capacity of PLZT decreases by about 20 per cent after cooling the sample from 200° C to room temperature in an electric field E = 4.0 kV/cm.

Applying an electric field (E \leq 4.3 kV/cm) at low temperatures no changes in either the power release $\dot{q}(E,T,t)$ or the heat capacity were detected.

4. Long-time heat relaxation was observed after an electric field was switched on or off at a certain temperature. As in the case of relaxation after cooling, t^{-1} dependence of $\dot{q}(E,T,t)$ after field change is transformed into an exponential one for large t. The maximum relaxation time τ_{max} is proportional to E; $\dot{q}(E,T,t)$ is proportional to E^2 and does not depend on T (1.1 K < T \leq 3K).

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19.	Biophysics

Салинг С., Колач М., Салинг А. Экспериментальное изучение влияния электрического поля на низкотемпературную медленную релаксацию в кристаллических сегнетоэлектриках

Были проведены калориметрические измерения поликристаллического Pbo, pisLao, oss(Zro, esTio, ss)0, при гелиевых температурах в электрическом поле E(0 \leq T \leq 4,3 кв/см). Тепловыделение после охлаждения от T₁(1,3K \leq T₁ \leq \leq 35K) до T₀ = 1,3K подобно наблюдаемому в аморфных металлах и диэлектриках. Экспериментальные результаты не согласуются со стандартной туннельной моделью. Наблюдаемое тепловыделение объясняется предположением существования максимальной энергии 'r в функции распределения. Максимальное время релаксации rmax зависит от T₁. Подобное тепловыделение наблюдалось после включения или выключения электрического поля, оно не зависит от T в области 1,1K \leq T \leq X и пропорционально E², а максимальное время релаксации пропорционально E. В монокристалле KH₂PO₄ тепловыделение не наблюдалось.

Работа выполнена в Лаборатории ядерных проблем ОИЯИ.

Препринт Объединенного института ядерных исследований. Дубна 1987

Sahling S., Koláč M., Sahling A. Experimental Study of Electric Field Influence on Low-Temperature Long-Time Relaxation in Crystalline Ferroelectrics

E8-87-499

Calorimetric measurements with polycrystalline Pbo.015Lao.085 $(2ro.65Tio.35)O_3$ were performed at helium temperatures in electric field E $(0 \le E \le 4.3 \text{ kV/cm})$. Heat release after cooling from $T_1(1.3 \text{ K} \le T_1 \le 35 \text{ K})$ to $T_0 = 1.3 \text{ K}$ is very similar to that in amorphous metals and dielectrics. Experimental results disagree with the standard tunneling model. The observed heat release may be explained assuming the existence of a maximum energy ϵ_f in the distribution function. The maximum relaxation time τ_{max} was found as a function of T_1 . A similar heat release is observed after switching on or off the electric field, independent of T for 1.1 K $\le T \le 3 \text{ K}$, proportional to E^3 with $\tau_{max} \sim E$. No heat release was observed in the KH_2PO_4 single crystal.

The investigation has been performed at the Laboratory of Nuclear Problems, JINR.

Preprint of the Joint Institute for Nuclear Research. Dubna 1987