

THERMAL EXPANSION OF THE LiKB_4O_7 AND $\text{CsLiB}_6\text{O}_{10}$ SINGLE CRYSTALS

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UDC 536.413
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The results of dilatometric investigations of the thermal expansion coefficients performed over a wide temperature range for LiKB_4O_7 and $\text{CsLiB}_6\text{O}_{10}$ single crystals are presented. It is shown that all the thermal expansion coefficients α_{ij} are positive for LiKB_4O_7 crystals, while some of them are negative for $\text{CsLiB}_6\text{O}_{10}$ ones. This can be explained with the use of the 'membrane' effect model that is typical of the corrugated and spiral structures. It is concluded that the temperature changes in a phonon spectrum also influences the character of the $\alpha_{ij}(T)$ dependences.

temperature, and these values markedly differ from each other.

This paper presents the results of the dilatometric studies carried out over a wide temperature range and the analysis of the data obtained. As an integral part, it includes the calculation of the temperature dependences of all the CLTE $\alpha_{ij}(T)$ as well as the coefficients of volumetric thermal expansion (CVTE) $\beta(T)$ for LiKB_4O_7 and $\text{CsLiB}_6\text{O}_{10}$ single crystals over the temperature ranges 80–870 and 87–930 K, respectively.

1. Introduction

Noncentrosymmetric single crystals of anhydrous borates are considered as promising materials for the higher-order harmonic generation of the laser emission, especially in the far ultraviolet spectral region. This concerns, in particular, the single crystals of $\text{CsLiB}_6\text{O}_{10}$ belonging to a tetragonal syngony (the $I\bar{4}2d$ space group) [1] and LiKB_4O_7 which belong to an orthorhombic syngony (the $P2_12_12_1$ space group) [2].

It was shown in papers [2,3] that, upon the substitution of a half of Li atoms by K ones, a new chemical compound which is melted congruently at the temperature $T_{\text{melt}} \approx 953$ K is formed in the $\text{Li}_2\text{B}_4\text{O}_7$ – $\text{K}_2\text{B}_4\text{O}_7$ system [3]. However, the physical properties of LiKB_4O_7 single crystals have not been studied at all.

A situation with $\text{CsLiB}_6\text{O}_{10}$ crystals is somewhat better, since much attention has been paid to the investigation of their optical parameters which are promising from the viewpoint of practical applications. However, other physical properties of these single crystals, especially a thermal expansion, have been studied insufficiently. Only two papers devoted to the study of the thermal expansion in a temperature range from room temperature to 500 °C by means of the X-ray [4] or dilatometric [5] method were published within this field. In both cases, however, values of the coefficients of linear thermal expansion (CLTE) α_a and α_c were calculated only for room

2. Experimental Technique

LiKB_4O_7 ($T_{\text{melt}} \approx 953$ K) and $\text{CsLiB}_6\text{O}_{10}$ ($T_{\text{melt}} \approx 1120$ K) single crystals were grown by the Czochralski method from the corresponding stoichiometric melts with the use of Pt-crucibles [6,7]. A growth charge for each of the compounds was obtained by the method of multistage temperature synthesis from carbonates K_2CO_3 , Li_2CO_3 , and Cs_2CO_3 and boric acid H_3BO_3 .

Since for the crystals of the orthorhombic syngony with the $P2_12_12_1$ space group, to which LiKB_4O_7 belongs, the α_{ij} CLTE tensor has three non-zero components α_{11} , α_{22} , and α_{33} (or α_a , α_b , and α_c , respectively), the measurements were carried out for all three directions, x , y , and z . The specimens, $9 \times 5 \times 4$ mm in size oriented along the main crystallographic axes x , y , and z were made of a grown LiKB_4O_7 single crystal.

For the crystals of the tetragonal syngony with the $I\bar{4}2d$ space group, to which $\text{CsLiB}_6\text{O}_{10}$ belongs, the α_{ij} CLTE tensor has only two non-zero components $\alpha_{11} = \alpha_{22} = \alpha_a = \alpha_{\perp}$ and $\alpha_{33} = \alpha_c = \alpha_{\parallel}$. That is why, it is enough to perform the measurements for two directions, x and z . The specimens of $11 \times 9 \times 6$ mm in size oriented along the main crystallographic axes x , y , and z were made of a $\text{CsLiB}_6\text{O}_{10}$ single crystal with especial caution: all operations of the mechanical treatment, namely cutting and grinding, were performed in such a way that a contact with water or even humid air was avoided. For this reason, the $\text{CsLiB}_6\text{O}_{10}$ single

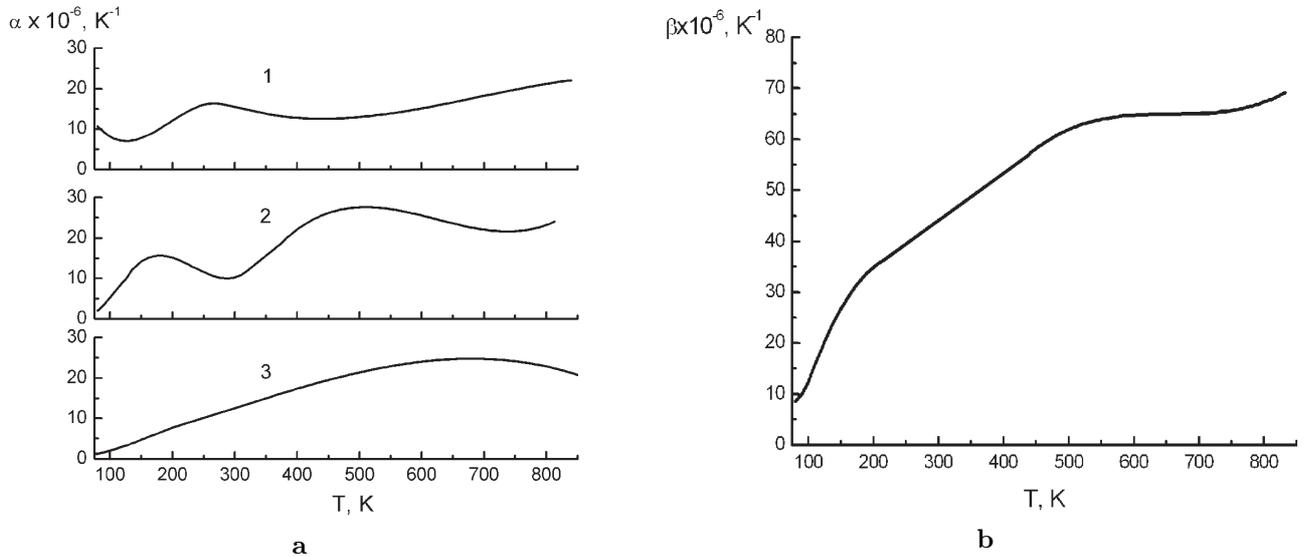


Fig. 1. Temperature dependences of the coefficients of thermal expansion for the LiKB_4O_7 single crystal: *a* – CLTE α_a (1), α_b (2), and α_c (3); *b* – CVTE

crystal, as well as all specimens made from it, were kept in a desiccator, and the surface was protected by oil during a treatment.

The investigations of the thermal expansion were carried out with the use of a quartz dilatometer containing the capacitive displacement indicator working with an accuracy of not worse than 1×10^{-9} m (for a specimen length of 17 mm) [8]. Such a value of the dilatometer sensitivity ensures also the reliable results of CLTE measurements for smaller specimens which were used in this work. The α_{ij} vs T curves were calculated by means of the computer differentiation of $\Delta L/L = f(T)$ dependences obtained experimentally with a step $\Delta T = 0.25$ °C. As a result, the precision of the α_{ij} determination was not worse than $\pm 0.2 \times 10^{-6} \text{ K}^{-1}$. To obtain the temperature dependences of CVTE, the calculations were performed according to the formula for the corresponding syngony: $\beta = \alpha_a + \alpha_b + \alpha_c$ for LiKB_4O_7 and $\beta = 2\alpha_a + \alpha_c$ for $\text{CsLiB}_6\text{O}_{10}$.

3. Results of Measurements

Both the $\alpha_{ij}(T)$ and $\beta(T)$ dependences obtained as described above are shown in Figs. 1 and 2 for LiKB_4O_7 and $\text{CsLiB}_6\text{O}_{10}$ single crystals, respectively. It is worth to note that the temperature changes of CLTE have a complicated character, and there is a sizeable difference between the $\alpha_{ij}(T)$ dependences within the temperature range under study. At the same time, as is seen from the figures, there are no abrupt jumps which could

give an unambiguous evidence for the structural phase transitions in LiKB_4O_7 and $\text{CsLiB}_6\text{O}_{10}$ single crystals.

For LiKB_4O_7 crystals, there are indistinct maxima and minima on the $\alpha_a(T)$ and $\alpha_b(T)$ curves within the temperature ranges 125–150, 260–275, and 450–500 K and near 740 K. What attracts attention is a quite good coincidence of the extremal points on the temperature scale where each maximum on the $\alpha_a(T)$ curve corresponds to a minimum on the $\alpha_b(T)$ one, and vice versa (Fig. 1, *a*). On the other hand, as it is characteristic of the majority of solids, the $\alpha_c(T)$ curve is monotonous over the whole temperature range and does not exhibit any peculiarities. Fig. 1, *b* shows the calculated temperature dependence of CVTE, $\beta(T)$, which has a monotonous character. There are shoulder-like features in the vicinity of temperatures 200, 500, and 800 K. At room temperature (293 K), the thermal expansion coefficients take the values $\alpha_a = 16 \times 10^{-6} \text{ K}^{-1}$, $\alpha_b = 11 \times 10^{-6} \text{ K}^{-1}$, $\alpha_c = 12 \times 10^{-6} \text{ K}^{-1}$, and $\beta = 39 \times 10^{-6} \text{ K}^{-1}$.

It is seen from Fig. 2, *a* that, for $\text{CsLiB}_6\text{O}_{10}$ crystals, the CLTE components differ not only in values, but also in a sign, since $\alpha_c < 0$ for almost the whole temperature range under study and particularly for $T > 100$ K. The more detailed examination of the character of changes shows that, according to the peculiarities of the CLTE behavior, the $\alpha_{ij}(T)$ curves can be divided into three temperature regions: 87–200 (region I), 200–700 (region II), and 700–930 K (region III). The specific

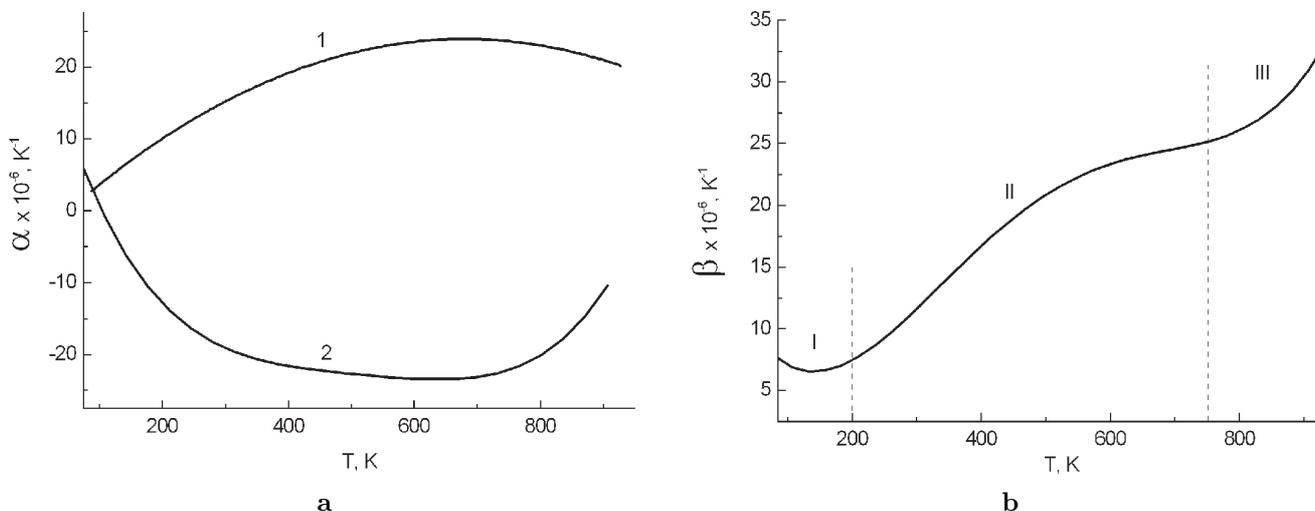


Fig. 2. The same is in Fig. 1 for a CsLiB₆O₁₀ single crystal: a CLTE – α_a (1) and α_c (2); b – CVTE

features characteristic of each region become apparent more clearly from Fig. 2, b, where the temperature dependence of CVTE, $\beta(T)$, is shown. As is seen from Fig. 2, b, a quick change of the α_a and α_c coefficients is observed, which is characteristic of regions I and III. However, these changes are almost equal and mutually compensated (β is almost unchangeable) for region I, but, for region III, the changes in absolute values of α_c are far more drastic. For region II, the temperature change in both the CLTE coefficients has a monotonous character.

For both single crystals LiKB₄O₇ and CsLiB₆O₁₀, we performed the calculations of the CLTE anisotropy index $\Delta\alpha$ that we introduced in work [8]. The $\Delta\alpha$ values calculated for a series of discrete temperatures are shown in Table 1. It is seen from the table that, for a LiKB₄O₇ crystal, when going from one temperature to another, the $\Delta\alpha$ values change chaotically, without exhibiting any signs of monotonicity. This is an additional argument that confirms the complexity of a character of the temperature changes of CLTE in these crystals. For CsLiB₆O₁₀ single crystals, however, $\Delta\alpha$ which equals $\alpha_{\perp} - \alpha_{\parallel}$ changes from zero at 90 K to $\approx 30 \times 10^{-6} \text{ K}^{-1}$ at 900 K, passing through a maximum with $\Delta\alpha \approx$

$\approx 48 \times 10^{-6} \text{ K}^{-1}$ at 600 K. These values of the CLTE anisotropy index are far smaller than those observed in LiB₃O₅, but, in average, they are close to the $\Delta\alpha$ values for β -BaB₂O₄. It is at low temperatures that the CLTE behavior becomes especially interesting: at first, α_a and α_c almost coincide at 90 K, taking on a value $\approx 2.8 \times 10^{-6} \text{ K}^{-1}$; then $\alpha_c(T)$ crosses zero and changes a sign to negative at 103 K. At room temperature (293 K), the thermal expansion coefficients get the values $\alpha_a = \alpha_{\perp} = 14.8 \times 10^{-6} \text{ K}^{-1}$ and $\alpha_c = \alpha_{\parallel} = -18 \times 10^{-6} \text{ K}^{-1}$ which are slightly different from those obtained by the authors of papers [4,5] ($\alpha_a = 20 \times 10^{-6} \text{ K}^{-1}$, $\alpha_c = -20 \times 10^{-6} \text{ K}^{-1}$ [4] and $\alpha_a = 21.2 \times 10^{-6} \text{ K}^{-1}$, $\alpha_c = -16.9 \times 10^{-6} \text{ K}^{-1}$ [5]). These discrepancies can result from the differences in measurement methods, features of the single crystal growth, or a specimen prehistory which depends, among other factors, on whether the CsLiB₆O₁₀ specimens have interacted with the moisture of the environment or not. It is unambiguously established that CsLiB₆O₁₀ single crystals can undergo hydration and hydrolysis, during which caesium ions can be even washed out from the channels of the structure. At the same time, upon heating, the dehydration processes occur and are finished at 130 °C [9]. The fact that these processes influence the properties, particularly optical ones, was noted in work [10].

The anisotropy coefficients $\Delta\alpha$ for the LiKB₄O₇ and CsLiB₆O₁₀ single crystals

$\Delta\alpha_i,$ 10^{-6} K^{-1}	LiKB ₄ O ₇					CsLiB ₆ O ₁₀				
	T, K					T, K				
	150	293	500	700	850	150	293	500	700	850
$\Delta\alpha_{a-c}$	2.5	4.0	-8.0	-7.0	1.5	13.5	32.8	43.9	47.9	40.2
$\Delta\alpha_{a-b}$	-4.5	5.0	-14.0	-4.0	-4.5					
$\Delta\alpha_{b-c}$	7.0	-1.0	6.0	-3.0	6.0					

4. Discussion of Results

LiKB₄O₇ single crystal. A characteristic feature, which differs LiKB₄O₇ single crystals from borate

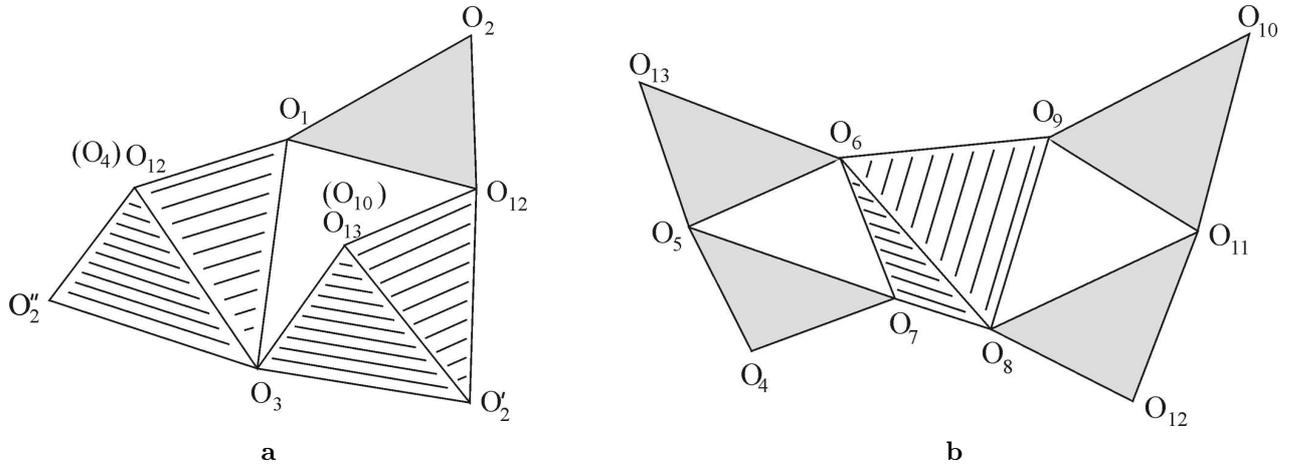


Fig. 3. Space configuration of the boron-oxygen complexes: *a* — $(\text{B}_3\text{O}_8)^{7-}$; *b* — $(\text{B}_5\text{O}_{10})^{5-}$

single crystals we studied earlier, namely BaB_2O_4 , LiB_3O_5 [8], and $\text{Li}_2\text{B}_4\text{O}_7$ [11], is the absence of the negative values of CLTE, especially in the temperature range 80–870 K. An explanation for such a temperature behavior of CLTE can be grounded on the fact that the structure of the LiKB_4O_7 crystal lattice differs from that of the above borates.

It was shown in work [2] that the crystal lattice of LiKB_4O_7 consists of anion boron-oxygen groups, which is typical of other borates. However, contrary to the case of BaB_2O_4 , LiB_3O_5 , or $\text{Li}_2\text{B}_4\text{O}_7$, where the frame structure of a crystal lattice is formed by only a single type of boron-oxygen complexes, LiKB_4O_7 contains two types of such groups, namely, the $(\text{B}_3\text{O}_8)^{7-}$ and $(\text{B}_5\text{O}_{10})^{5-}$ ones. Whereas the $(\text{B}_3\text{O}_8)^{7-}$ complex anion group consists of simple boron-oxygen complexes [one trigonal $(\text{BO}_3)^{3-}$, with a boron atom surrounded by three oxygen atoms, and two tetrahedral ones, where a boron atom is surrounded by four oxygen atoms], the $(\text{B}_5\text{O}_{10})^{5-}$ group consists of four $(\text{BO}_3)^{3-}$ and one $(\text{BO}_4)^{5-}$ complexes. In both of these anion groups, the simple complexes form six-membered rings — one ring in $(\text{B}_3\text{O}_8)^{7-}$ and two in $(\text{B}_5\text{O}_{10})^{5-}$ (Fig. 3). As is evident from the figure, the $(\text{B}_3\text{O}_8)^{7-}$ group (see Fig. 3, *a*) is actually an incomplete tetraborate boron-oxygen complex [12], while the $(\text{B}_5\text{O}_{10})^{5-}$ group consists of two three-borate complexes, with a simple tetraborate complex $(\text{BO}_4)^{5-}$ in-between [13]. The $(\text{B}_3\text{O}_8)^{7-}$ groups are bound with each other through a common oxygen atom and form infinite chains directed predominantly in the [100] crystallographic direction. The $(\text{B}_5\text{O}_{10})^{5-}$ groups, in their turn, are also interconnected with the $(\text{B}_3\text{O}_8)^{7-}$ ones, but through three common oxygen

atoms. An analysis shows that each unit cell of the LiKB_4O_7 crystal lattice shares four $(\text{B}_5\text{O}_{10})^{5-}$ groups and four $(\text{B}_3\text{O}_8)^{7-}$ ones. Any other variant, theoretically possible, could only be realized when a ratio between these two types of anion groups in the LiKB_4O_7 unit cell were 1 : 9, i.e. when it could contain one $(\text{B}_5\text{O}_{10})^{5-}$ groups and 9 $(\text{B}_3\text{O}_8)^{7-}$ ones, which is certainly unreal.

Thus, the origin of such a CLTE behavior, which, being characterized by the absence of negative values, is anomalous for a borate crystal, can be the presence of the $(\text{B}_5\text{O}_{10})^{5-}$ anion groups in a unit cell of LiKB_4O_7 , with these groups being arranged within the boron-oxygen frame of a crystal so that to counterbalance the influence of spirality of the above infinite chains of the $(\text{B}_3\text{O}_8)^{7-}$ groups.

There are additional data that serve as a confirmation of the fact that, in the LiKB_4O_7 single crystals, the influence of spirality of the chains of $(\text{B}_3\text{O}_8)^{7-}$ groups is absent. To these belong the results of the investigation of the propagation velocity of longitudinal acoustic waves along the directions of main crystallographic axes. So, along the *x* axis which, as is concluded in [2], is the axis of the aforementioned spirals, the value of velocity proves to be $V_{11} = 6460$ m/s. The corresponding values along other axes are $V_{22} = 4320$ m/s and $V_{33} = 5570$ m/s. However, as follows from the acoustic studies on other borates, for the crystallographic directions where the influence of spirality (or “corrugation”) of the boron-oxygen chains is noticeable, the velocity of acoustic waves is, as a rule, far smaller and rapidly decays. For example, in $\text{Li}_2\text{B}_4\text{O}_7$ single crystals where the axis of the double spirals of boron-oxygen complexes coincides with the direction of

the crystallographic axis z , the value of velocity achieves only $V_{33} = 5036$ m/s, whereas, for the perpendicular directions, the corresponding values exceed 7000 m/s ($V_{11} = 7358$ m/s and $V_{22} = 7460$ m/s). In the case of LiKB_4O_7 , however, the velocity of longitudinal acoustic waves along the axis of boron-oxygen spirals turn out to be higher than that for the other directions.

For this reason, the complicated and non-monotonous character of the $\alpha_a(T)$ and $\alpha_b(T)$ dependences in LiKB_4O_7 crystals is thought to originate from their high sensitivity to the temperature changes of a phonon spectrum. Unfortunately, the investigations of the Raman scattering spectra and the analysis of the phonon spectra in LiKB_4O_7 have not been carried out yet. However, with taking into account the complicated structure of the crystal lattice containing two types of boron-oxygen complexes and two kinds of cations, as well as the reduced symmetry of a unit cell, it can be reliably suggested that the phonon spectrum in LiKB_4O_7 is very complicated and has a nontrivial dependence on temperature.

The $\text{CsLiB}_6\text{O}_{10}$ single crystal. The presence of negative values of CLTE for the crystallographic direction z (see Fig. 2) makes it possible to attribute $\text{CsLiB}_6\text{O}_{10}$ single crystals to borate crystals with a typical CLTE behavior. Though the structure of the $\text{CsLiB}_6\text{O}_{10}$ crystal lattice is closer to that of the $\text{Li}_2\text{B}_4\text{O}_7$ one because it belongs to the tetragonal syngony, the character of the $\alpha_a(T)$ and $\alpha_c(T)$ curves observed in the $\text{CsLiB}_6\text{O}_{10}$ crystal is almost analogous to that characteristic of a LiB_3O_5 crystal. This means that, though the substitution of a half of Li atoms in LiB_3O_5 by Cs ones gives rise to a change of the syngony from the orthorhombic to tetragonal one, the crystal lattice of $\text{CsLiB}_6\text{O}_{10}$ remains analogous to that of LiB_3O_5 on a microstructural level, only with a somewhat smaller packing factor. For this reason, some authors consider $\text{CsLiB}_6\text{O}_{10}$ crystals as those belonging to nanoporous materials [9].

For $\text{CsLiB}_6\text{O}_{10}$ single crystals, the explanation of the temperature behavior of CLTE is possible by drawing an analogy with the behavior of the single crystals of other borates, particularly, by the use of a similarity with the structural features of the crystal lattice of LiB_3O_5 . The crystal lattice of $\text{CsLiB}_6\text{O}_{10}$, just as that of LiB_3O_5 , contains the three-dimensional circular boron-oxygen groups $(\text{B}_3\text{O}_7)^{5-}$ which consist of two $(\text{BO}_3)^{3-}$ complexes and one $(\text{BO}_4)^{5-}$ tetrahedron [13]. By means of a covalent bond, these complexes are bound into the infinite zigzag (corrugated) chains through the common (bridge-like) atoms of one apex of a BO_3 triangle and

one vertex of a BO_4 tetrahedron. So, when the above chains are elongated along the spatial diagonals of a unit cell of $\text{CsLiB}_6\text{O}_{10}$, their aggregations look like spirals in the direction of the z axis. Thus, the crystal lattice of $\text{CsLiB}_6\text{O}_{10}$, as in the cases of $\text{Li}_2\text{B}_4\text{O}_7$ and LiB_3O_5 , can be attributed to the chain-spiral structures, where a frame is formed by $(\text{B}_3\text{O}_7)^{5-}$ boron-oxygen complexes. By means of a covalent bond between the common oxygen atoms, these complexes are bound into spirals along the z axis, and the atoms of Cs and Li occupy the hollows formed in this frame. Thus, to interpret our results of the CLTE measurements for $\text{CsLiB}_6\text{O}_{10}$ single crystals, we can use the experience accumulated on the examination of the spiral and corrugated crystal structures, the characteristic of which is the negative values of at least one of the α_{ij} coefficients [15,16].

In $\text{CsLiB}_6\text{O}_{10}$ single crystals, as is seen from Fig. 2, *a*, for the directions perpendicular to z axis ($\alpha_a = \alpha_\perp$), the CLTE values remain positive over the whole temperature range under study (87–930 K). On the other hand, along the z direction, the α_c CLTE takes on negative values in the temperature range 103–930 K. Though the absolute value of α_c at some temperatures exceeds that of α_a , the CVTE remains positive over the whole temperature range under study (see Fig. 2*b*), as it is typical of the absolute majority of single crystals.

It is thought that, for $\text{CsLiB}_6\text{O}_{10}$ single crystals, as in the case of the crystals of other borates, there are two mechanisms leading to the negative values of α_c . The former one is caused by a corrugated structure [15], for which a specimen size depends not only on a distance between atoms, but also on the angle between the directions of bonds between them. As a result, a small increase in the bond length gives rise to a significant decrease in the angle between the bonds. With a change in temperature, this leads to a reduction of the size in one direction occurring simultaneously with its expansion in a perpendicular one. The latter mechanism originates from a strong narrowing of the acoustic branch of the lattice oscillation spectrum upon a temperature change, widely observed in the spiral structures [16], which results in the so-called “membrane” effect typical of the crystals with spiral and layered structures [17]. Since the negative values of α_c are practically observed in the whole temperature range under study, this means that, in $\text{CsLiB}_6\text{O}_{10}$, both the mechanisms act simultaneously and in one direction. The rapid reduction in the absolute values of both CLTE components at elevated temperatures (higher than 750 K), as shown in Fig. 2, *a*, can be explained by the mutual compensation of the increasing contributions of the usual

mechanisms of a thermal expansion, the phonon and libration ones.

5. Conclusions

1. In the single crystals of a novel borate LiKB_4O_7 , all the coefficients of thermal expansion are positive in the temperature range 80–870 K, which can be explained by the fact that the structure of its crystal lattice differs from that of other borates. The nonmonotonous character of the $\alpha_{ij}(T)$ dependences is brought about by a high sensitivity of these coefficients to the temperature changes of the phonon spectrum.

2. For $\text{CsLiB}_6\text{O}_{10}$ single crystals, the negative sign of one of the CLTE components, α_c , can be explained with the use of the 'membrane' effect model which is typical of corrugated and spiral structures.

3. To minimize the temperature-induced stresses in LiKB_4O_7 and $\text{CsLiB}_6\text{O}_{10}$ single crystals, arising in the process of post-growth cooling to room temperature, a special attention should be paid to the temperature intervals where $T < 700$ K, for which a strong anisotropy and a complicated character of the $\alpha_{ij}(T)$ dependences are observed.

4. The hygroscopicity of $\text{CsLiB}_6\text{O}_{10}$ single crystals can give rise to the inaccuracy in CLTE measurements.

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Received 15.09.05.

Translated from Ukrainian by A.I. Tovstolytkin

ТЕПЛОВЕ РОЗШИРЕННЯ МОНОКРИСТАЛІВ LiKB_4O_7 І $\text{CsLiB}_6\text{O}_{10}$

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Резюме

Наведено результати дилатометричних досліджень коефіцієнтів теплового розширення монокристалів LiKB_4O_7 і $\text{CsLiB}_6\text{O}_{10}$ у широкому температурному інтервалі. Встановлено, що всі коефіцієнти теплового розширення LiKB_4O_7 додатні, а α_c для $\text{CsLiB}_6\text{O}_{10}$ має також і від'ємні значення, що пояснено із застосуванням моделі "мембранного" ефекту для гофрованих і спіральних кристалічних структур. Зроблено висновок, що на характер залежностей $\alpha_{ij}(T)$ в цих кристалах впливають також температурні зміни фононного спектра.