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The growth of $\text{Li}_2\text{B}_4\text{O}_7$ single crystals and investigation of low temperature elastic anomalies

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ABSTRACT : The growth installation and peculiarities of growth $\text{Li}_2\text{B}_4\text{O}_7$ (LBO) crystals by Czochralsky method have been described in this paper. Experimental results of temperature dependences of ultrasonic velocities for various modes and crystallographic directions were measured and some anomaly in this dependence was discovered. Our results are discussed in comparison with some previous data.

LBO is very promising piezoelectric material due to high electromechanical properties, founding on the temperature compensated cuts and low cost of starting materials. High quality LBO single crystals were grown by both Czochralsky (Cz) [1] and Bridgman (Br) [2] methods.

For LBO growth we used special apparatus which was designed in ICRAS. These installations allow to easily change furnaces and pulling mechanism (depending on needed temperature of the melt, kind and pressure of gaseous medium and growth method). Parameters of chamber are shown in Table 1, ones of pulling mechanism in Table 2. Using these chambers, pulling mechanisms and different furnaces, we can quickly prepare various configurations of Cz and Br.

Table 1 : Parameters of growth chambers.

	CROT-M	CROT-I	CROT-B
Height (mm)	450	600	630
Int. diameter (mm)	260	350	440
Work pressure (Pa)		2-60000	

These chambers consist of three parts which can be easily separated for motion seeds and furnaces.

Table 2 : Pulling and rotation mechanisms for crystal or crucible.

	A	B
Max. pulling range (mm)	150	300
Pulling velocity (mm/hour)	0.1-9.9	0.1-9.9
Rotation velocity (rpm)	15-99	5-99

While growing LBO two peculiarities have most importance : relatively small density, that requires high precision of weight sensor, and stoichiometric composition of melt.

In first experiments were used LiOH , Li_2CO_3 , B_2O_3 , H_3BO_3 as starting materials. The best results were obtained with Li_2CO_3 and H_3BO_3 . Crystals were grown from platinum crucibles on air and

from graphite crucibles at neutral atmosphere with $\langle 100 \rangle$ or $\langle 001 \rangle$ oriented seeds. Pulling rate was equal to 0.7 mm/hour, rotation - 50 rpm. The axial gradient near melt was 40 K/cm, radial gradient - 10 K/cm. The rate of cooling of crystal in growth chamber was 20 K/hour at the first stage and 50 K/hour at the last one.

Crystal pulling and rotation mechanism (its parameters are pointed in Table 2) include weight sensor and precise pulling rate gauge. This allows to keep constant diameter of growing LBO crystal up to 80 mm if using 100 mm crucible with enough precision.

Crystals grown under optimal conditions were optically transparent, did not include visible defects and had low mechanical stress. But if grown from nonstoichiometric compound, these crystals sometimes became un transparent while cooling in the growth furnace.

Elastic, piezoelectric and dielectric parameters of our crystals at room temperature according to paper [3], devoted to comparative analysis of various author's data, are the most reliable results and not differ from our results of best crystals.

Absolute value of difference between a lot of measured results at room temperature is relatively small. But the view of low temperature dependences of sound velocities sharply differs not only from one author to another, but also in different experiments.

We consider this difference is connected with presence of incommensurate phase under thermocycling, as it is supposed in [4], although existence of incommensurate phase is denied in [5] from NMR experiments and the results of [4] are explained by jumps of charge due to pyroeffect.

We repeated our previous experiments on measuring temperature dependences of sound velocities on the electrically short-circuited crystals, when the influence of pyroeffect may be neglected. On the diagrams one can see that there are same jumps of sound velocity approximaty at the same temperatures, as well as with open crystals. In this experiment, thermocycles were repeated many times and temperature points of such jumps slightly varied within range $-165..-145^{\circ}\text{C}$.

Figure : Temperature dependence of the shear elastic wave velocity propagated along the z-axis LBO. The inset shows the dependence of the temperature of the jump in velocity value on the number cooling cycle.

So, wide application of LBO crystals requires new additional investigations of behavior of different parameters at low and high temperatures.

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