

# TECHNIQUE FOR GROWTH OF CsI:Tl CRYSTALS WITH INTERNAL RADIOISOTOPE FOR NUCLEAR PHYSICS

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CsI:Tl crystals have been grown by Stockbarger technique in sealed quartz ampoules. Grown ingots do not adhere to the material of the container. Ready crystals have no residual stresses and do not break during subsequent mechanic treatment. Vibration absorption of grown crystals contains no bands of OH<sup>-</sup> or CO<sub>3</sub><sup>2-</sup> ions in the IR region. After irradiation the electronic absorption has no bands of F- or F<sub>A</sub>-color centers in the visible region. Spectrometric characteristics of the grown crystals are not inferior to standard. Developed technique is recommended for growth of CsI:Tl scintillation material with internal source of radiation. The peculiarity of the method is the dehydration of raw material at T ≤ 40°C under conditions which exclude photolysis of salt.

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## INTRODUCTION

CsI:Tl crystal is well known inorganic scintillation material which widely used in scientific and industrial applications [1]. Such scintillators with radioactive isotopes, uniformly distributed over the crystal lattice are used in nuclear physics experiments [2] for soft X-rays registration that accompany the β-decay of radioisotopes especially in case of K-capture. It has been shown [3] that events of extremely low energy 0.84 keV from <sup>22</sup>Na internal source are reliably registered while such events have not available for external sources recording.

The process of obtaining such crystals involves some difficulties since no harmful substances are allowed to enter production facilities. Open technique, for instance Kyropoulos technique, is not used for crystal growth in this case due to environmental safety. Ordinarily, crystal with internal isotopes is grown by Stockbarger technique in sealed quartz ampoules. There is a fundamental limitation for crystal growth in quartz ampoule which connected with procedure of surface melting of ready ingot. Violation of ampoule impermeability during surface melting process results in pollution of industrial rooms by radiation. If you skip this step, the lateral surface of ingot will adhere to the wall of ampoule and grown crystal will crack during cooling. The diameter and height of crystal is larger, the probability of cracking is higher.

It is well known that the interaction the ingot with quartz is due to presence of hydroxide ions in the melt and crystal [4, 5]. Developed growth techniques [6] to prevent pollution of the melt by OH<sup>-</sup> and CO<sub>3</sub><sup>2-</sup> ions, including additional treatment of melt by different cation [7, 8] to transfer oxygen-containing impurities into insoluble compounds. The disadvantage of these methods is unavoidable pollution of melt administered cations [7] and the associated negative effect on the functional characteristics of the crystals.

In this regard, the actual task is to provide a growth technique that allows obtain CsI:Tl scintillation material without oxygen-containing impurities, eliminates interaction of the ingot with the walls of quartz ampoule and no effect on the scintillation characteristics of ready detectors.

## EXPERIMENTAL PROCEDURE

For crystal growth we used cesium iodide producing by Sigma-Aldrich Corporation. Before experiments each part of salt was tested on the presence of so-called "high-temperature water" [4]. Methods such verification using thermal desorption of gases is described in [9]. Only those parts of salt were selected for crystal growth which did not show the peaks of water desorption above 180°C during the heating process.

Crystals were grown of two types, which differs the method of preparation of raw materials. First of all it was the crystals of CTT (Traditional Technology) type which corresponds to the ordinary drying of cesium iodide. The ampoule was filled with salt in the industrial room at a temperature of 20°C and 60% relative humidity under luminescent lighting. Filled ampoule was pumped out to a residual pressure of 5·10<sup>-3</sup> Torr and then evacuated for 30 min. Then the furnace temperature is increased to T = 560°C with the rate of 50°C/h. Ampoule with salt prepared in this manner considered ready for crystal growth.

For the crystals of second type (CNT – New Technology) drying of salt was carried in the same way. The difference is that all the operations for filling ampoules by wet salt, its storage and preparation of raw materials were carried out under red light to prevent photolysis. Ampoules were 50 mm in diameter and 180 mm in height, since at such size generally not possible to avoid the adhesion of the ingot to the quartz using ordinary process of salt dehydration.

After drying, the crystals were grown by the Stockbarger technique in the same quartz ampoules. The melt was not pumped. Directional crystallization was carried out by lowering the ampoule at a rate of 2 mm/hour.

Crown ingots were cut on a slides and samples of 10×10×10 mm size were manufactured for spectrometric characteristics measuring. Polishing of ready scintillator was carried out with the advice of [10, 11] to minimize the damaged layer and prevent the formation of dead layer. Measuring the light output was carried out on the spectrometry stand [12] with a photodiode Hamamatsu S3590-08 type with respect to the standard. The accuracy of light output measuring was ±3.2% [13].

For afterglow measuring the samples of  $\varnothing 20 \times 4$  mm sizes were used. Measurements of afterglow level were carried out using NPG3 device. Anode current of X-ray tube was of 0.6 mA at a voltage of 140 kV. The afterglow level was determined at 100 ms after excitation pulse finishing. The duration of the X-ray pulse was equal to 1 s.

## RESULTS

The most sensitive test for the presence of  $\text{OH}^-$  ions in the melt and grown crystal [4, 5] is the nature of wetting the walls of the ampoule and sticking of ingot to the material of container. Unlike ingots of CTT the grown CNT crystals did not stick to quartz and easily extracted from the ampoule. They showed low level of residual stresses and did not crack in the further machining.

The fact that adhesion of the ingot to the material of the container is not observed means that the crystals are free of hydroxide ions. The analysis of the vibration absorption spectra of grown crystals fully confirms this conclusion. In case of CNT there are any absorption bands in infrared part of spectrum. It is well known that oxygen-containing impurities like  $\text{CO}_3^{2-}$ ;  $\text{OH}^-$  first of all are characteristic for ingot grown in air atmosphere and also  $\text{IO}_3^-$ ;  $\text{SO}_4^{2-}$ ;  $\text{NO}_3^-$ ;  $\text{SiO}_3^{2-}$  sometimes [5].

Contrary to CNT, weak absorption bands are observed in CTT crystals. The spectrum is very similar to spectrum presented in [7] for large-sized CsI crystals non-activated and doped by  $\text{Eu}^{2+}$ . Well known that noted absorption bands correspond to stretching and deformation vibrations of  $\text{CO}_3^{2-}$  ions [14, 15]. Using the relationship [16] between absorption coefficient and concentration of these ions we estimated the quantity of carbonate ions ( $C_K$ ) and concluded that  $C_K \leq 5 \cdot 10^{-5}$  mole % in CTT crystal.

**Spectrometric characteristics.** In view of the small size of the ingot, the study of spectrometric characteristics was carried out for samples measuring  $10 \times 10 \times 10$  mm cut from the beginning of the cylindrical part of the ingot, where thallium is almost uniformly distributed throughout the volume. We can assume a priori that noted quantities of impurities cannot adversely affect the light output and energy resolution of CsI:Tl crystal, since the concentration of major impurity (thallium) is a lot more and it equal to  $C_{\text{Tl}} \approx 1.05 \cdot 10^{-1}$  mole %. This value has been determinate for the layer of ingot corresponding to a sample of  $10 \times 10 \times 10$  mm size.

In Fig. 1 the pulse height spectrum of photodiode detector made from CTT crystal is presented. For convenience of comparison the data on scintillation characteristics are summarized in the table for CsI:Tl crystals of two types. The light yield and energy resolution for CsI:Tl standard also presented in this table.

Indeed as it follows from the data of the table that the light yield of all grown crystals corresponds to the standard. The energy resolution of the produced photodiode detectors within the error of measurements does not differ from the standard and is equal to  $R = 4.82\%$  for CTT and  $R = 4.87\%$  for CNT. It should be noted that the measurements were carried out for a fixed shap-

ing time of  $t_{RC} = 10 \mu\text{s}$ . Comparison of these results with the data of [17], where was published one of the best value  $R = 4.84\%$  for crystals of  $1 \text{ cm}^3$  size with a photodiode, indicates a high spectrometric quality of crystals of both types.

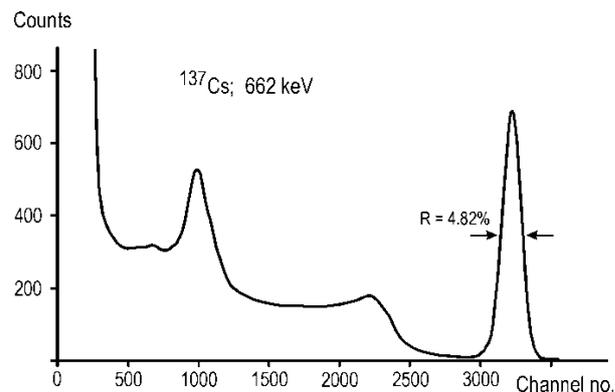


Fig. 1. Pulse height spectrum of photodiode detector made from CsI:Tl of CTT-type. Excitation by  $^{137}\text{Cs}$  source, crystal size is  $10 \times 10 \times 10$  mm, photodiode Hamamatsu S3590-08

An important problem of photodiode scintillator is the possibility of detecting low-energy quanta. So another illustration of spectrometric quality of grown crystals is data for X-ray with energy of 59.6 keV from  $^{241}\text{Am}$ . The corresponding pulse height spectrum is shown in Fig. 2 for the CNT material.

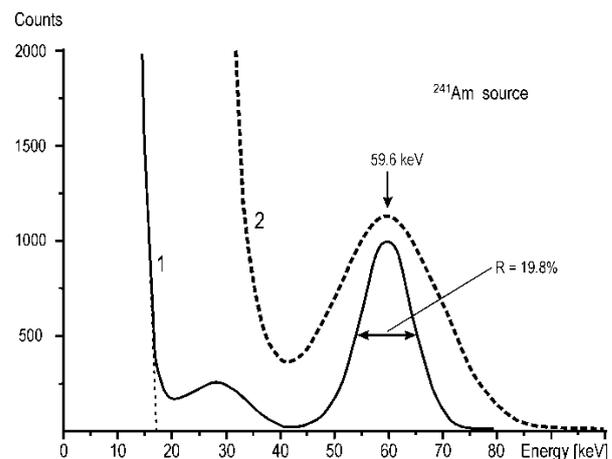


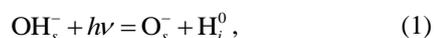
Fig. 2. Pulse height spectra of photodiode detectors made from CsI:Tl of CNT-type. Used photodiodes are FD-05-25 (1) and S3590-08 (2). Excitation by  $^{241}\text{Am}$  source

A distinctive feature of the spectrum in Fig. 2 is that in this case the Ukrainian photodiode FD-05-25 [18] with sensitive area of  $4 \times 4$  mm was used. In Fig. 2 the abscissa axis is not shown in the channels, but replaced by an equivalent energy scale, this was done for the convenience of observing the shift in the registration threshold. It can be seen from data of curve 1 that the peak of the total absorption is well distinguished, the energy resolution for line of 59.6 keV is 19.8%, and the detection threshold is reduced to 17 keV in comparison with the threshold of  $\sim 40$  keV for curve 2, where the pulse height spectrum presented for similar crystal ( $1 \text{ cm}^3$ ) coupled to photodiode with a sensitive area  $10 \times 10$  mm.

The data given on the energy resolution and detection threshold of grown crystals in the X-ray range do not differ from the best known values for scintillator based on CsI:Tl crystals, described, for example, in Ref. [17].

From analysis of data presented in the table we can conclude that, as we assumed a priori, spectrometric quality of CNT material do not differ from CTT crystal. It means that grown crystals are not inferior in performance to well known scintillation materials CsI:Tl, CO<sub>3</sub> and perspective CsI:Tl, NO<sub>2</sub> [17].

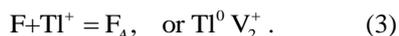
**Radiation hardness.** In contrast to the light output, even negligible quantity of OH<sup>-</sup> and CO<sub>3</sub><sup>2-</sup> impurities have a sharply negative effect on the radiation hardness of CsI:Tl crystals. It is known that these crystals colored easily under daylight, when they contain noted impurities. Coloration is due to specific radiation-chemical reactions in the crystal lattice. The essence of the mechanism [19] consists in that under action of radiation or irradiation of ultraviolet light OH<sup>-</sup> is decomposed by the reaction:



where the subscripts *s* and *i* denote the position of a particle at the lattice site or interstitial, respectively. Due to the high mobility of the H<sub>i</sub><sup>0</sup> atom, it easily diffuse by the interstices and reacts with carbonate ions according to the reaction:



where V<sub>A</sub><sup>+</sup> is an anion vacancy. According to reaction (2) divalent carbonate ion transforms to monovalent hydrocarbonate, and an anion vacancy converted into F-center. In CsI:Tl crystal formed F-centers are located near the cation of Tl<sup>+</sup> and create so-called F<sub>A</sub>-centers consisting of anion vacancies, activator cation and electron [15, 20]:



Designation F<sub>A</sub> assumes electron localization mainly on the anion vacancy, and (Tl<sup>0</sup> V<sub>A</sub><sup>+</sup>) – on cation of thallium [20].

Absorption coefficient [cm<sup>-1</sup>]

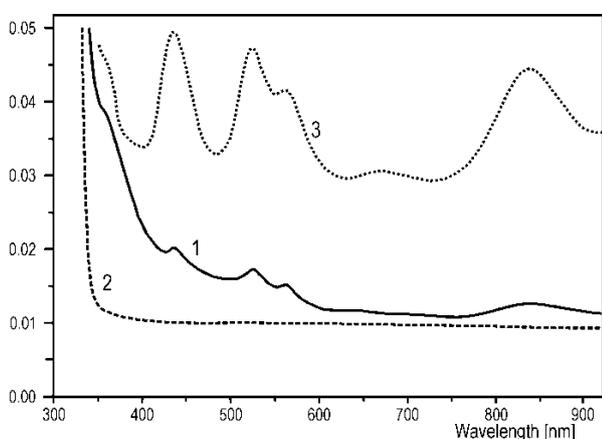


Fig. 3. The absorption spectra of irradiated crystals of CTT-type (1) and CNT-type (2). 3 – Absorption spectrum of CsI:Tl, CO<sub>3</sub> crystal from [21]

The absorption spectra of grown crystals in visible range of spectrum are shown in Fig. 3. Curve 2 corresponds to the absorption of CNT irradiated by dose of 10 Gy. It is seen that in contrast to the CTT (curve 1) F<sub>A</sub> color centers are not formed according to the above-described mechanism in the CNT. Small values of the absorption coefficient before irradiation (*k*<sub>0</sub> ~ 0.01 cm<sup>-1</sup>) indicate a high quality of both materials as well as the quality of surface treatment.

It should be noted that curve 1 corresponds to the best of the three grown crystals of CTT type. Despite the relatively small changes in *k*, the induced absorption bands are clearly seen in the spectrum.

For the convenience of identifying the peaks, curve 3 is also shown in Fig. 3, according to the data of [21]. This curve corresponds to the absorption of CsI:Tl, CO<sub>3</sub> crystal. It should be noted that the curve 3 characterizes induced absorption (*k* – *k*<sub>0</sub>) in the irradiated crystal and in Figure is shifted up. As it seen from a comparison of curves 1 and 3, the absorption maxima of the irradiated CTT material fully coincide on position with bands of CsI:Tl, CO<sub>3</sub> crystal.

Another structurally sensitive parameter of crystals is the storage of energy in impurity traps. It is known that the peak of thermally stimulated luminescence (TSL) at 300 K and the minute afterglow of CsI:Tl crystals at room temperature are associated with the presence of OH<sup>-</sup> and CO<sub>3</sub><sup>2-</sup> ions [16]. TSL curves for crystals of two types are presented in Fig. 4. Curve 1 corresponds to energy storage in CTT material and curve 2 to CNT. It is clear seen that intensity of TSL at 300 K for the CNT sample is ten times weaker than the CTT. According to the data of [16, 21] a minute afterglow at room temperature is associated with intensity of TSL peak at 300 K while the two components of millisecond afterglow are closely connected with peaks at 180 and 215 K [21]. The nature of the last two peaks is associated with contamination of the raw material with Me<sup>2+</sup> cation. From a comparison of curves 1 and 2, it can be seen that the proposed drying process significantly reduces the intensity of TSL in the 300 K region and does not affect the intensity of the peaks at 180 and 235 K. The data on the afterglow level after 100 ms and 10 min are given in the table and are in full accordance with the results just described.

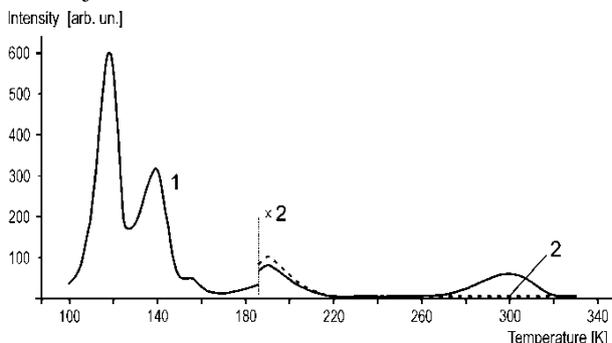


Fig. 4. The TSL curves of irradiated at 77 K crystals of CTT-type (1) and CNT-type (2)

## DISCUSSION

So, it is established that the proposed technique of crystal growth, including preliminary selection of salt

and its dehydration, allows obtain CsI:Tl crystal of high-quality without co-activator like CO<sub>3</sub>. It is shown that grown CNT crystals did not stick to quartz and easily extracted from the ampoule. We believe that the problem of crystal growth without surface melting has been solved.

However, the high values of scintillation characteristics in themselves are not direct proof of the advantages of proposed drying technique. The fact is well known [4] that quartz can provide a deoxidizing effect on the melt. Prolonged exposure of the melt leads to the following chemical reaction in the liquid phase:



In our conditions, water may evaporate from the melt; condense on the cold parts of the ampoule and not to participate in further reactions. Silicates are concentrated on the walls of ampoules, forming patterns on the interface, similar to the frost. In this case grown ingot do not contains hydroxyl ions but nevertheless interacts with ampoule. Such situation is typical for the case of "pure" crystal growth from "dirty" melt.

Such melt cleaning by the material of ampoule itself becomes ineffective when growing large crystals. We have already noted that recently more effective scavengers and getters are found [8].

It should be recalled that grown ingots of CNT type do not sticking to the wall of the quartz ampoule. This fact just completes the results on scintillation characteristics and gives the proof on the absence of OH<sup>-</sup> ions in the melt and ingot. The source of material contamination by noted impurities is a drying process itself in case of growth CTT material. It should be noted that we do not observe the release of "high temperature water" from selected raw material. An ampoule with salt prepared to crystal growth sometimes was cooled to room temperature and subjected to additional hydration. Such

sample testing shows that gas desorption above 180°C take place only for CTT samples not for CNT one.

The lack of interaction between the ingot and quartz allows excluding the operation of surface melting. Last circumstance improves the environmental safety of the process. This method of crystal growth is recommended to obtain CsI:Tl ingot with internal radionuclide for application in nuclear physics [2], geology and radioecology monitoring [22].

## CONCLUSIONS

CsI:Tl crystals have been grown by Stockbarger technique in sealed quartz ampoules. Obtained ingots do not adhere to the container material, have no residual stresses and not break down during subsequent machining.

Spectra of IR absorption don't show the presence of bands which are connected with OH<sup>-</sup> and CO<sub>3</sub><sup>2-</sup> ions. Electronic absorption in visible range doesn't show the bands of F- and F<sub>A</sub>-centers. Spectrometric characteristics of the grown crystals are not inferior to standard.

The lack of interaction between the ingot and quartz ampoule allows excluding the operation of surface melting. Last circumstance improves the environmental safety of the process. Such technique of crystal growth is recommended to obtain CsI:Tl ingot with radioisotope for application in nuclear physics.

A reliable criterion of salt suitability to obtain the ingot without impurities like OH<sup>-</sup> and CO<sub>3</sub><sup>2-</sup> is the lack of high-temperature desorption peak of water. It is shown that a necessary condition for the preparation of the salt is thoroughly dehydrated at a temperature no higher than 40°C with the exclusion of photolysis.

*Characteristics of crystals in comparison with standard*

	Characteristics	CsI:Tl crystals		
		Standard	CTT	CNT
1	Interaction of ingot with quartz	–	yes	no
2	Bands of IR absorption, cm <sup>-1</sup>	no	880; 1390	no
3	Light yield, photon/MeV	48 000	48 700	48 200
4	Energy resolution, %	4.90	4.82	4.87
	on <sup>137</sup> Cs (10×10×10 mm)			
	on <sup>241</sup> Am (4×4×4 mm)		19.8	19.2
5	Registration threshold, keV	17	17	17
6	Level of afterglow, %	0.8	0.55	0.56
	after 100 ms		0.07	< 0.005
	after 10 min			
7	Color centers after D = 10 Gy	no	F <sub>A</sub>	no

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## ПОЛУЧЕНИЕ КРИСТАЛЛОВ CsI:Tl С ВНУТРЕННИМИ РАДИОНУКЛИДАМИ ДЛЯ ЯДЕРНОЙ ФИЗИКИ

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Методом Стокбаргера в герметичных кварцевых ампулах выращены кристаллы CsI:Tl, которые не прилипают к материалу контейнера, не имеют остаточных напряжений и не разрушаются при последующей механической обработке. Спектры колебательного поглощения выращенных кристаллов CsI:Tl не содержат полос поглощения в ИК-области спектра, обусловленных ионами  $\text{OH}^-$  и  $\text{CO}_3^{2-}$ , а электронное поглощение в видимой области после облучения не имеет полос поглощения центров окраски F- и  $F_A$ -типов. Спектрометрические характеристики выращенных кристаллов не уступают эталону. Метод рекомендован для выращивания сцинтилляторов с изотопами, введенными в кристаллическую решетку, и отличается дегидратацией сырья при  $T \leq 40^\circ\text{C}$  в условиях, исключающих фотолиз соли.

## ОТРИМАННЯ КРИСТАЛІВ CsI:Tl З ВНУТРІШНІМИ РАДІОНУКЛІДАМИ ДЛЯ ЯДЕРНОЇ ФІЗИКИ

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Методом Стокбаргера в герметичних кварцевих ампулах вирощено кристали CsI:Tl, які не прилипають до матеріалу контейнера, не мають залишкових напружень і не руйнуються в процесі подальшої механічної обробки. Спектри коливального поглинання отриманих кристалів CsI:Tl не мають смуг поглинання в ІЧ-області спектра, що обумовлені іонами  $\text{OH}^-$  і  $\text{CO}_3^{2-}$ , а електронне поглинання у видимій області після опромінення не мають смуг поглинання центрів забарвлення F- і  $F_A$ -типів. Спектрометричні характеристики вирощених кристалів не поступаються еталону. Метод рекомендовано для вирощування сцинтиляторів з ізотопами, що введені до кристалічної ґратки, і відрізняється дегідратацією сировини при  $T \leq 40^\circ\text{C}$  в умовах, що виключають фотолиз солі.