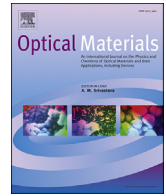




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## Development and melt growth of novel scintillating halide crystals

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## ABSTRACT

Melt growth of scintillating halide crystals is reviewed. The vertical Bridgman growth technique is still considered as very popular method that enables production of relatively large and commercially attractive crystals. On the other hand, the micro-pulling-down method is preferable when fabrication of small samples, sufficient for preliminary characterization of their optical and/or scintillation performance, is required. Moreover, bulk crystal growth is also available using the micro-pulling-down furnace. The examples of growths of various halide crystals by industrially friendly melt growth techniques including Czochralski and edge-defined film-fed growth methods are also discussed. Finally, traveling molten zone growth that in some degree corresponds to horizontal zone melting is briefly overviewed.

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## 1. Introduction

A number of a metal halide crystals is known to be a very efficient scintillating materials. Therefore, much efforts and resources were recently applied to develop technologies of their crystal growth from the melt.

Regarding simple halide compounds, three main groups are generally considered with respect to scintillating solid-state substances. (1) The alkali halides (AHa) are formed by alkali metals (A) of Li, Na, K, Rb, and/or Cs, and halogen elements (Ha) of F, Cl, Br, and/or I. These materials behave as highly stable crystalline compounds. In most cases, they can be produced directly from the melt of stoichiometric composition [1,2]. As a result, tens of tons of alkali halide scintillating crystals are produced yearly on commercial basis [3]. These compounds are the most used scintillating materials. They favorably combine good detection efficiency, high light

output and low production cost. (2) The scintillating alkali-earth halides (AeHa<sub>2</sub>) are formed by alkaline earth (Ae) metals of Ca, Sr, and/or Ba and one of the halogens (Ha). These compounds are also well-known scintillating substances when they are activated by Eu<sup>2+</sup>, especially, after the rediscovery of Eu-doped SrI<sub>2</sub> (Eu:SrI<sub>2</sub>) by Cherepy et al. [4]. Eu:SrI<sub>2</sub> is recognized as one of the highest performance scintillator with high light yield and high energy resolution. (3) The rare-earth halides (LnHa<sub>3</sub>) represent third group of simple compounds formed by lanthanide metals (Ln) and halogen elements [5]. Moreover, there is a number of complex halide crystals that include either solid solution compounds [6,7] or complex halides having a structure different from those of the source simple halides [8–11]. Regarding solid solution crystals, some of them are produced by mixing of two isostructural end members, however others are simply doped crystals [6].

The purpose of this review is to summarize recent progress in the development of the melt growth techniques suitable for the production of halide crystals and their application as scintillators. The growth of fluoride crystals is not discussed here because the Ce-doped iodides, bromides, and chlorides are more efficient

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scintillators as compared to fluoride compounds that produce less scintillation light [12,13]. The high light yield of such halides is greater than that of the corresponding fluorides because of their smaller band-gap. Moreover, the energy resolution of fluoride scintillators is usually worse compared to that of the other halides (Fig. 1).

## 2. Starting materials processing

Many of halide compounds are extremely hygroscopic [5,14]. This makes the crystal growth process and corresponding growth hardware relatively complicated and brings various problems even at the stages preceding the crystal formation. Therefore, even the procedures performed at room temperature before the growth itself require an additional care. These procedures include handling of raw materials, their mixing, charging the crucible, seed setting, etc. As an example, the loading of starting materials of KI, SrI<sub>2</sub>, CaI<sub>2</sub>, and EuI<sub>2</sub> into the crucibles [10,15] used for the growth of corresponding halide crystals was performed in dry box containing less than 0.1 ppm of moisture and oxygen. Moreover, the growths themselves were also performed in well-controllable and moisture-free atmosphere that was established by sealing the crucible/ampoule before its heating.

Post-growth treatment of the as produced crystals also needs additional efforts: all the procedures of crystal cutting, polishing, and following characterization must be performed in the well-isolated glove boxes to avoid destruction of the samples due to their interaction with moisture present in the ambient atmosphere and following decomposition. Without such precautions, the crystals and especially their surfaces degrade with formation of solid and/or liquid layers that have composition and structure different from the grown crystal. Definitely, such degradation of

properties affects the scintillation performance of the crystals [14]. However, in some cases, repolishing of the surface of the halide crystals (KCaI<sub>3</sub>:Eu) that were exposed to the ambient atmosphere for the short period of time is effective [10]. In such a way, in the case of fortunate circumstances, the scintillation performance of the specimens can be reestablished. However, exposure of the samples to air atmosphere for several hours or days leads to their hydrolysis under formation of different oxidic impurities such as hydroxo- and oxo-halides, and carbonates and thus may result in their complete decomposition. Therefore, the crystals are often stored in mineral oil or under dry and inert atmosphere environment. In such conditions, the quality of the crystals and their surfaces do not change for a long time. Nevertheless, removal of oxidic impurities from starting materials before their crystal growth is of high importance to prevent the grown crystal from sticking to container wall and its cracking. Application of different chlorination agents (e.g. HCl, COCl<sub>2</sub>, and Cl<sub>2</sub>), which react and remove oxidic impurities, by introducing those directly into molten alkali and lead halides was reported in Refs. [16] and [17], respectively. Similarly, Sakuragi et al. [18] presented the development of Liquinert process by applying thermal treatment under vacuum on SrI<sub>2</sub>:Eu in combination with reactive-gas atmosphere processing.

Another technique that allows protection of the mixture of the starting materials and the grown crystals during all the procedure from their possible reaction with moisture and/or oxygen is based on the application of removable growth chambers. Such chambers can be transported from the glove box used at the pre-growth stages of the process preparation to the growth unit without direct contact of the crucible and the raw materials with surrounding ambient atmosphere [19,20]. This innovation was experimentally tested for the vertical Bridgman (VB), the micro-pulling-down ( $\mu$ -PD), and Czochralski (CZ) growths discussed below. In such designs, all the pre-growth procedures including setting of the crucible, its charging, and preliminary installation of the seed are performed in the glove box under high purity inert atmosphere. Typically, concentrations of oxygen and moisture in such environment do not exceed 1 ppm [19,20]. Thereafter, the removable chamber is isolated from the atmosphere of the glove box and then it is transported to the growth system without direct contact of starting materials with external (room) environment. When the chamber is attached to the growth machine, it is connected to the vacuuming and gas supplying systems again to establish atmosphere necessary for the growth. Schematic diagram and view of actual removable chamber applicable for the VB growth are illustrated in Fig. 2 (at room temperature). The view of such a type of chamber with heated crucible during the growth is demonstrated in Fig. 3. In the growth systems with removable chamber, direct sealing of the crucible, as it is practiced in the case of the quartz crucibles/ampoules, is not necessary. After the completion of growth the removable chamber with the crucible that contains the as-grown crystal is isolated from the growth system again and then it is transported back to the glove box for the following after-growth operations. The advantage of the removable chamber technology is the fact that there is no need of quartz ampoule sealing, which is limited by maximum 1.5 inch in diameter so far. Consequently, SrI<sub>2</sub>:Eu single crystal with 2 inch in diameter is grown by the removable chamber technology using halide micro-pulling down (H- $\mu$ -PD) furnace (Micropuller H-01, C&A Corp.) [20].

## 3. Vertical Bridgman growth

The VB method (Fig. 4) is one of the most widely used techniques applied for fabrication of halide crystals. To some extent, the VB growth system arrangement is rather simple [3]. The starting solid material is charged into the growth container (crucible). It

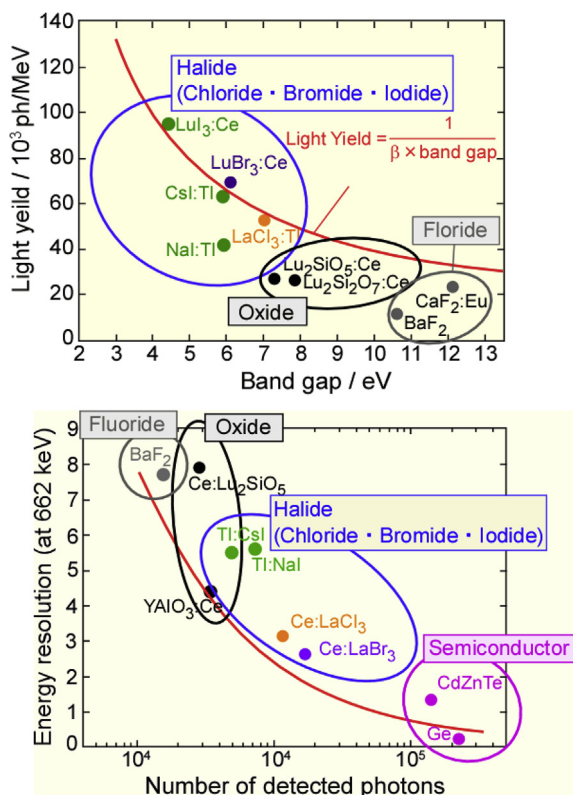


Fig. 1. Light yield (top) and energy resolution (bottom) of various scintillating materials according to [12,13].

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