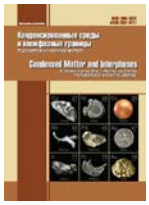

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Growth and physical properties of $CaSrBaF_6$ single crystals

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Abstract

Using the Bridgman-Stockbarger method, crystals of triple fluoride CaF_2-SrF_2-BaF_2 were grown in a composition range similar to that of $CaSrBaF_6$. The crystals were 10-12 mm in diameter and 50–60 mm in length. The $CaSrBaF_6$ crystal is a new optical material which is transparent in the mid-IR, visible and UV ranges. The uneven distribution of the components along the length of the crystal did not exceed 10 %. The edge of the absorption band in the IR range was 14.3 μm, and the optical absorption at the wavelength of 200 nm did not exceed 18 % (less than 0.2 cm^{-1}). The refraction indices were 1.4527, 1.4488, and 1.4458 for the wavelengths of 633, 969, and 1539 nm respectively. The crystal melts in the temperature range of 1150–1210 °C. The $CaSrBaF_6$ composition is an appropriate matrix for doping with rare-earth ions in order to obtain functional single-crystal and ceramic materials of the visible and IR ranges.

Keywords: Calcium fluoride, Strontium fluoride, Barium fluoride, Fluorite, Solid solution, Isomorphism, High entropy alloys

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1. Introduction

Calcium, strontium, and barium fluorides crystallise in the fluorite structure with the following parameters of the crystal lattice: 5.463, 5.800, and 6.200 Å respectively. Single crystals of difluorides of alkaline earth elements are widely used as photonics materials [1–3] as well as matrices for doping with rare-earth ions [4-10]. They are characterised by wide transmission regions from vacuum ultraviolet to the mid-IR range. However, the use of pure fluorides can be limited when designing optical systems [11]. The use of solid solutions allows varying the physical properties and characteristics of matrices over a wide range. Continuous areas of solid solutions with the valleys on the melting curves are formed in the CaF_2–SrF_2 [12, 13] and SrF_2–BaF_2 systems [14, 15]. We grew and studied the corresponding series of single crystals $Ca_{1-x}Sr_xF_2$ and $Sr_{1-x}Ba_xF_2$ [16–22]. Isomorphism in the CaF_2–BaF_2 system is limited [18, 23, 24]. The corresponding binary solid solutions are of interest as optical materials for photonics. When isovalent solid solutions are formed, physical properties of the crystals significantly change (compared to the components), including the refractive index [16–18, 20], vibration spectra [25], and hardness [17, 20]. On the whole, mechanical characteristics of solid solutions improve, thermal conductivity decreases, and electrical conductivity increases. Spectral-luminescent characteristics and cluster structure of doping REE change in a non-monotonic way.

In recent years, multicomponent phases with several isostructural elements in their composition have been attracting greater interest. Such compositions containing 5 and more components were called high-entropy alloys (HEAs) [30, 31]. According to the third law of thermodynamics, these single-phase alloys cannot be stable at low temperatures, although only slow processes of atomic diffusion and phase relaxation in some cases help to reveal their kinetic stability and potential applications. Homogeneous materials with multicomponent compositions are usually found in glass [32]. Initially, this term had been used for metal alloys, but then HEA oxides were also found [33]. The synthesis of high-entropy fluoride ceramics $CeNdCaSrBaF_{12}$ was reported [34].

The purpose of this work was to grow single crystals of the triple-component solid solution $Ca_{1-x-y}Sr_xBa_yF_2$ similar to the $CaSrBaF_6$ composition and to study its properties. The corresponding composition can serve as a matrix for doping with rare-earth ions and obtaining a multicomponent functional material.

2. Experimental

We used shards of CaF_2 (OST 3-6304-87) and BaF_2 optical single crystals together with the remelted SrF_2 powder (extra-pure grade) as the starting substances to grow CaF_2–SrF_2–BaF_2 crystals. It is preferable to choose crystal reagents along with powder that was remelted under fluorinating atmosphere, as the reagents do not absorb moisture and can be stored for a long time. Each initial reagent was controlled by differential scanning calorimetry (DSC), X-ray diffraction analysis (XRD), and electron microscopy.

We grew the crystals of triple fluoride CaF_2-SrF_2–BaF_2 in the composition range similar to $CaSrBaF_6$ on an automated system NIKA-3 under conditions of induction heating of a six-cell graphite crucible placed inside the inductor. The temperature gradient was formed using graphite pipes and disks as screens that had radial sawcuts to exclude the heating with the induction current, which allowed obtaining the temperature gradient (according to the temperature of crucible wall) of about 30 °C/cm. The temperature was measured through the chamber windows using a manual IR pyrometer. As soon as the crucible was filled with the mixture, pumping was performed to the residual pressure of no more than $5 \cdot 10^{-2}$ mbar. The CF_4 gas that partially filled the chamber was used as a fluorinating agent. After that, it was smoothly heated (for 1.5–2 hours), and when the operational temperature was reached, the crucible was removed from the hot area to the cold area at 6 mm/hour. When the removal process was finished, the crucible was slowly cooled for 4–6 hours.

We performed a thermal analysis of the crystals on a Netzsch DSC 404 F1 differential scanning calorimeter. The measurements were made in platinum crucibles in a flowing argon atmosphere. Ground fragments of the seed boule cone were used as samples. We performed thermal analysis of all the crystals in the range of

temperatures of 20-1400 °C in the mode of two heating-cooling cycles.

The refractive index of the samples of the crystals was measured on a Metricon 2010 refractometer. The measurement method was based on the determination of the critical angle of incidence at which light starts going into the volume of the sample through the surface of the measuring prism (similar to an Abbe refractometer). This device allows performing measurements at three wavelengths: 633, 969, and 1539 nm. The measurements were performed on the crystal samples with the polished side surface in the region of 5–10 mm from the seed boule cone.

Spectrophotometers Shimadzu UV-2600 and Infralyum FT 02 were used to register optical transmission in the UV, visible, and IR ranges of the optical spectrum. The measurements were taken using a dual-beam method in the UV and visible range and using a single-beam method in the IR range. The measurements were performed on the samples with two polished side surface in the region of 5–10 mm from the seed boule cone.

The elemental composition of the crystals was studied on a Quanta 200i 3D FEI scanning electron microscope with the system of energy dispersive X-ray microanalysis which included an Apollo X energy dispersive silicon detector with a resolution of > 131 eV for an MnK line at 100000 imp/s. The peak-to-background ratio was no less than 10000/1. The concentration of the components of the crystals was measured in three regions along the crystal's length at the distances of 1 mm, 20 mm, and 40 mm from the seed boule cone. Three measurements were taken at different points of each region, and then the results were averaged.

3. Results and discussion

We grew a series of crystals that were 10–12 mm in diameter and 50–60 mm in length (Fig. 1). The crystals were optically transparent (Fig. 2). The uneven distribution of the components of the crystal along the length of the boule did not exceed 10 % for most of the crystals. The most uniform distribution was observed on the crystal of the $CaSrBaF_6$ composition (33 mol % CaF_2 – 33 mol % SrF_2 –33 mol % BaF_2), Fig. 3.

The DSC curves for the sample of the crystal of the $CaSrBaF_6$ composition for the first heating-cooling cycle are presented in Fig. 4. The sample melts in the range of temperatures of 1150–1210 °C.

The results of the measurement of the refractive index are presented in Table 1. The maximum values of the refractive index are typical for the sample 31 mol % CaF_2 – 31 mol %

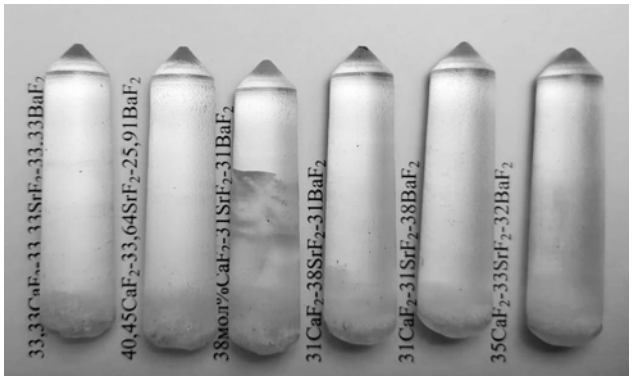

Fig. 1. Photo of untreated boules of triple fluoride CaF_2–SrF_2–BaF_2 crystals in the composition range similar to $CaSrBaF_6$

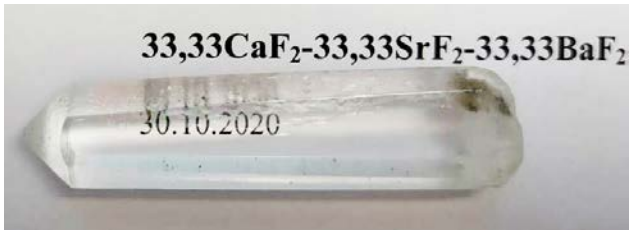

Fig. 2. Photo of a polished triple fluoride $CaSrBaF_6$ crystal

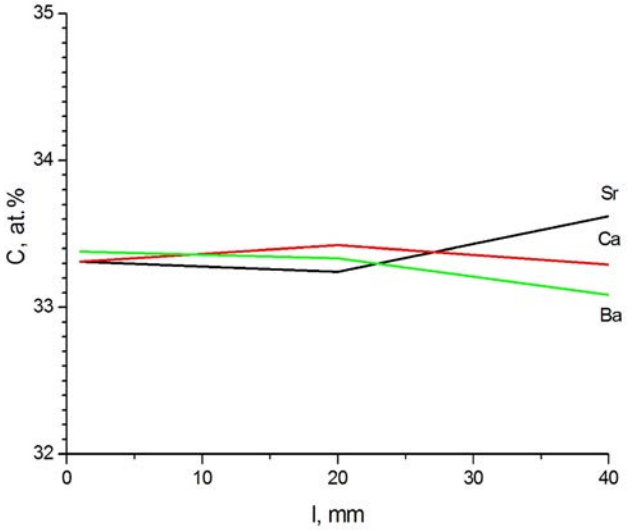

Fig. 3. Distribution of the components of a $CaSrBaF_6$ crystal along the length of the boule for the 33 mol % CaF_2 – 33 mol % SrF_2 – 33 mol % BaF_2 composition

Table 1. Values of the refractive index *n* at three wavelengths for crystals of triple fluorides in the composition range similar to $CaSrBaF_6$

Compositions	λ = 633 nm	λ = 969 nm	λ = 1539 nm
33 mol % CaF_2 – 33 mol % SrF_2 – 33 mol % BaF_2	1.4527	1.4488	1.4458
40.5 mol % CaF_2 – 33.6 mol % SrF_2 – 25.9 mol % BaF_2	1.4497	1.4458	1.4430
38 mol % CaF_2 – 31 mol % SrF_2 – 31 mol % BaF_2	1.4522	1.4483	1.4451
31 mol % CaF_2 – 38 mol % SrF_2 – 31 mol % BaF_2	1.4520	1.4472	1.4448
31 mol % CaF_2 – 31 mol % SrF_2 – 38 mol % BaF_2	1.4566	1.4526	1.4491
35 mol % CaF_2 – 33 mol % SrF_2 – 32 mol % BaF_2	1.4527	1.4486	1.4451

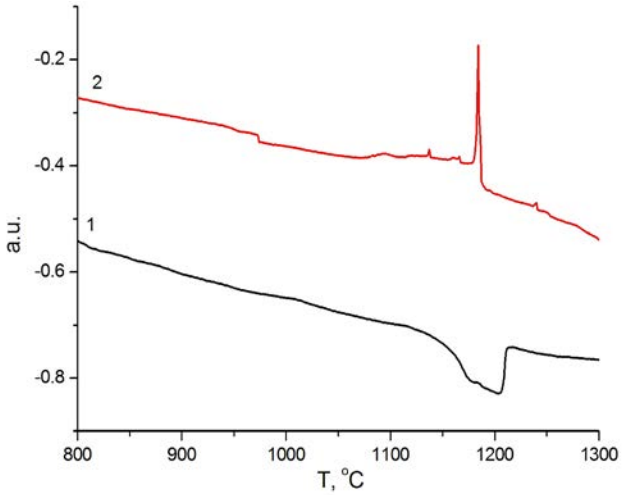

Fig. 4. Sections of the DSC curves of a crystal sample of the 33 mol % CaF_2 – 33 mol % SrF_2 – 33 mol % BaF_2 composition, first cycle: *1* – heating, *2* – cooling

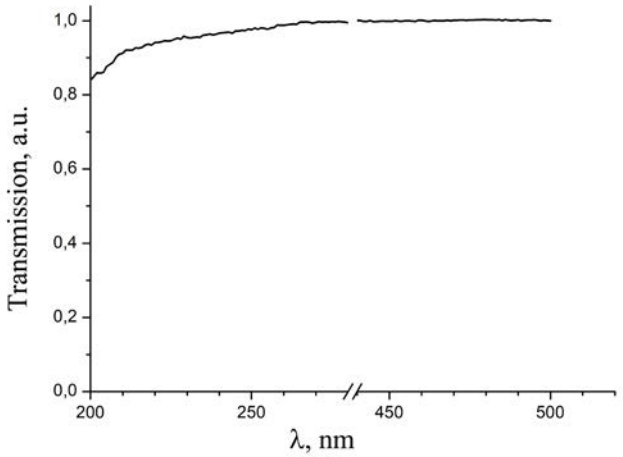

Fig. 5. Transmission spectrum of a crystal sample of the 33 mol % CaF_2 – 33 mol % SrF_2 – 33 mol % BaF_2 composition in the UV and visible range. The thickness of the sample is 10 mm

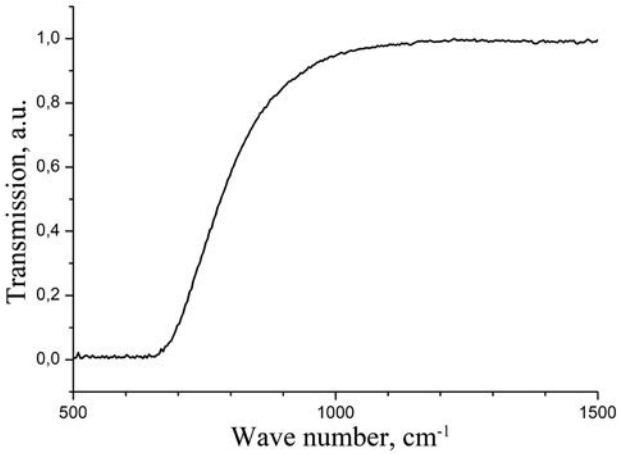

Fig. 6. Transmission spectrum of a crystal sample of the 33 mol % CaF_2 – 33 mol % SrF_2 – 33 mol % BaF_2 composition in the IR range. The thickness of the sample is 10 mm

SrF_2 – 38 mol % BaF_2 while the minimum values are typical for the composition 40.5 mol % CaF_2 –33.6 mol % SrF_2 – 25.9 mol % BaF_2.

The transmission spectra for the crystal of the $CaSrBaF_6$ composition in the region of UV and IR absorption edges are presented in Fig. 5 and Fig. 6 respectively. The measured sample was 10 mm thick. The spectra are presented taking into account the Fresnel reflection from the surfaces of the sample. The edge of the UV absorption was beyond the operating area of the spectrophotometer, and the absorption at the wavelength of 200 nm did not exceed 18 % (less than 0.2 cm^{-1}).

The border region of the IR absorption for the transmission degree of 0.1 begins from 700 cm^{-1} (14.3 μm). 50 % transmission occurred at 12.5 μm.

Therefore, the crystal of the $CaSrBaF_6$ composition is a new optical material which is transparent in the mid-IR, visible, and UV ranges. A big difference between the temperatures of liquidus and solidus exceeding 50 °C is indicative of the incongruent nature of the melting of this composition. Consequently, the growth from the melt of $CaSrBaF_6$ crystals of high optical quality which are suitable for laser applications can hardly be implemented due to the problems

with concentration overcooling, instability of the crystallisation front, and the formation of a cellular and dendritic substructure [35, 36]. However, this composition can be a suitable crystal matrix for obtaining upconversion luminophores [37] and can be used in the production technology for optical ceramics [38].

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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