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Effects of co-doping with divalent cations on performance of YAG:Ce,C scintillator

I. Gerasymov, S. Witkiewicz-Lukaszek, T. Zorenko, Yu. Zorenko, D. Kofanov, Ya. Boyaryntseva, S. Tkachenko, P. Arhipov, E. Galenin, D. Kurtsev, O. Zelenskaya, V. Alekseev, K. Lebbou, O. Sidletskiy

Abstract— Growth technologies of oxide crystals in W/Mo crucibles have been developed as a low-cost alternative to conventional processes involving Ir crucibles. Carbon-containing atmosphere needed to protect crucibles from oxidation leads to introduction of carbon into the crystal lattice, creation of carbon-related defects, which affect scintillation performance. Meanwhile, a search for fast scintillators for new generation of positron-emission tomographs and high energy physics experiments at colliders is under way. Codoping with divalent cations has become an efficient way to suppress long components of scintillation decay in Ce-doped scintillators. This work addresses $Y_3Al_5O_{12}$ (YAG) crystals codoped with carbon, cerium activator and divalent cations. Optical and scintillation properties of YAG:Ce,C,A²⁺ crystals (A=Ca²⁺, Mg²⁺, Ba²⁺, Sr²⁺) are systematically studied. YAG:Ce,C,Ca²⁺ demonstrated the fastest scintillation decay times, which are promising for the mentioned applications. Mechanisms of scintillation process in the studied materials are discussed. The Ce³⁺/Ca²⁺ ratio in YAG:Ce,C,Ca²⁺ was optimized to minimize slow components in scintillation decay.

Index Terms— Codoping, crystal growth, scintillator, yttrium aluminum garnet, W crucible

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I. INTRODUCTION

CODOPING with mono- or divalent cations is a common strategy for enhancing performance of Ce³⁺-doped scintillators. There are positive examples of the energy resolution enhancement in LaBr₃:Ce,Sr [1], whereas scintillation decay acceleration and afterglow suppression were reported in Ce-Mg²⁺- or Ce-Ca²⁺-codoped complex oxides such as rare earth orthosilicates (LSO), garnets (YAG, LuAG, GAGG) [2-6], and perovskites (YAP) [7]. Mg²⁺-codoping of GAGG:Ce, the brightest oxide scintillator, was proposed for detectors at the upgraded LHCb calorimeter where a main decay time of up to 15 ns and a light yield of at least 15000 are targeted [8].

However, providing a fast timing while keeping the light yield on a reasonable level is challenging. Introduction of mono- or divalent cations into trivalent cation sites creates an excessive negative charge that may be compensated by Ce³⁺ transfer into the tetravalent state, while the increased concentration of single Ce⁴⁺ centers provides a faster radiative de-excitation [3]. Meanwhile, Ce⁴⁺-Mg²⁺ pairs with a lower barrier for thermal quenching from the 5d₁ excited state of cerium at heavy Mg²⁺-doping contribute to thermal quenching of Ce³⁺ scintillation yield [9]. Therefore, acceleration of the scintillation decay time in garnets at heavy Mg²⁺-doping is achieved at the expense of light yield [8].

Recently, we have introduced the crystals of carbon-containing garnets, obtained as a result of crystal growth in carbon-containing atmosphere. Some enhancement of light yield with carbon doping was reported, while timing characteristics were similar [10] or slower [11] as compared to garnets obtained from Ir crucibles under conventional conditions of neutral atmosphere. In the same works, the light yield enhancement in YAG:Ce,C up to ca. 30000 phot/MeV, LuAG:Ce,C and LuYAG:Ce up to ca. 27000 phot/MeV was attributed to the creation of negatively charged carbon-containing defects, prevailing over intrinsic defects, such as oxygen vacancies or antisites responsible for color centre formation and deterioration of scintillation performance in garnets. According to first principles simulations [12, 13], V_O (oxygen vacancy), C_O (carbon occupying oxygen site) and V_{Al} (aluminum vacancy) are the most probable among the isolated defects in as-grown crystals at crystal growth under reducing conditions of CO-containing atmosphere. However, the lowest formation energies are obtained for the C_O-V_{Al4} and C_i-Y_{Al} complex defects (V_{Al4} is an aluminum vacancy in a tetrahedral

site; Y_{Al} is yttrium occupying Al sites) . These defects in positive oxidation states may trap electrons thus forming different types of color centers. The situation completely changes after annealing in air. The lowest formation energies are obtained for $C_{Al}-V_O$, C_O-V_{Al} and C_i-V_{Al6} defects stabilized in negative oxidation states, which do not capture electrons and eventually do not form color centers. This presumably causes irreversible discoloration of C-doped garnets after thermal annealing [14] and a good radiation hardness (in other words, weak deterioration of transmission) under high doses of gamma-rays and high-energy protons [15]. Furthermore, at our best knowledge, there are no reports on systematical study of the impact of divalent cations on scintillation and optical performance.

This work addresses triple codoping of YAG:Ce,C with divalent cations aiming to reduce the scintillation decay time while keeping a high enough yield. Such challenge thus targeting the requirements to scintillators for upcoming LHCb collider experiment [8]. Therefore, we have chosen Mg^{2+} , Ca^{2+} , Sr^{2+} , Ba^{2+} divalent cations and studied the optical and scintillation performance of YAG:Ce,C crystals codoped with these elements.

II. EXPERIMENTAL

A. Crystal growth and sample fabrication

The process of obtaining garnets by the Czochralski method from W crucibles in Ar+CO atmosphere is described in detail elsewhere [16]. The starting components in the form of Y_2O_3 , Al_2O_3 , CeO_2 , CaO , MgO , SrO , BaO with the purity of not less than 4N were mixed at the stoichiometric ratio and melted in W crucibles of 50 mm x 50 mm size. The crystals with a diameter of 10-15 mm and cylinder height of 60-70 mm were pulled from melt with a rate of 1-2 mm/hour with automated diameter control by a weight sensor. The samples of 5 mm x 5mm x 2 mm and 5 mm x 5mm x 1 mm dimensions were cut and polished for tests of optical and scintillation properties. The nominal Ce content in melt was 0.3 at.% in respect to Y, and the contents of Sr, Ba, Mg, Ca were the same. Additionally, Ce and Ca contents in YAG:Ce,Ca were varied to optimize the Ce/Ca ratio. Then the samples were annealed in air at 1250 °C for 24 hours to reveal the role of thermal annealing on optical and scintillation parameters.

B. Characterization

The absorption spectra were registered using a Jasco 760 UV-Vis spectrometer in the 200–1100 nm range. The luminescence decay curves under UV-Vis excitation were measured using a combined fluorescent lifetime and steady-state spectrometer FLS 920 (Edinburgh Instruments) equipped with hydrogen filled nF 900 ns flashlamp (optical pulse duration is 1.0–1.6 ns, pulse repetition rate is 40 kHz) for time correlated single photon counting measurements. The CL spectra were registered using a SEM JEOL JSM-820 electron microscope equipped with a Stellar Net spectrometer and TE-cooled CCD detector working in the 200–925 nm range.

Express control of the scintillation light yield and

scintillation decay kinetic of film parts of composite scintillators and substrates as well was determined using the set-up consisting of a Hamamatsu H6521 photomultiplier, homemade multi-channel analyzer working with the shaping time of 12 μ s, and a Tektronix TDS3052 digital oscilloscope under excitation by α -particles of ^{239}Pu (5.15 MeV) source. As α -particle penetration depth in the studied materials is approximately 12-15 μ m the spectra of the studied bulk crystals might be compared with the standard YAG:Ce film sample with a photoelectron yield of 360 phels/MeV and a LY of 2.65 photons/KeV.

As the measurements under α -particles reflect the state of just the sample surface of few tens micrometer thick, additional scintillation decay measurements were performed at ISMA NAS of Ukraine under γ -radiation (^{137}Cs , 662 keV). Signal from Hamamatsu R6231 PMT anode was transferred to the input of Rigol DS6064 19 oscilloscope. The absolute light yield was determined by comparing the peak position at pulse height spectra of our crystals and of BGO crystal (8600 ph./MeV with an energy resolution of 11.9%). The measurements were recorded using a pulse processing chain consisting of a R1307 PMT (Hamamatsu, Japan), a charge-sensitive preamplifier BUS 2-95 24 (Tensor, Russia), a custom shaping amplifier and a multichannel analyser AMA-03F (Tensor, 25 Russia). ^{137}Cs (662 keV) was used as a gamma-ray source.

Scintillation decay time spectra were measured with Hamamatsu R6231 PMT excited with 662 keV gamma ray from ^{137}Cs source. Signal from PMT anode was fed to the input of Rigol DS6064 19 oscilloscope.

All the measurements except thermostimulated luminescence (TSL) were performed at room temperature (25 °C).

III. RESULTS AND DISCUSSION

A. Optical properties of YAG:Ce,C,A²⁺ crystals

The photos of crystals with the different codopants obtained by the Czochralski method are presented in Fig. 1. The as-grown crystals were transparent with a grey-brownish tint. The boules did not contain visible inclusions in the bulk except for the lowest part grown before the cutoff from the melt. Upper parts of the crystal lateral surfaces were etched due to interaction with the growth atmosphere (see [16] for the details).

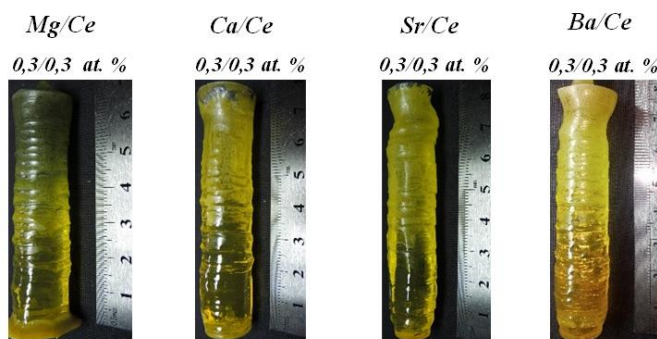


Fig. 1. As-grown YAG:Ce,C,A²⁺ crystals obtained in the present work.

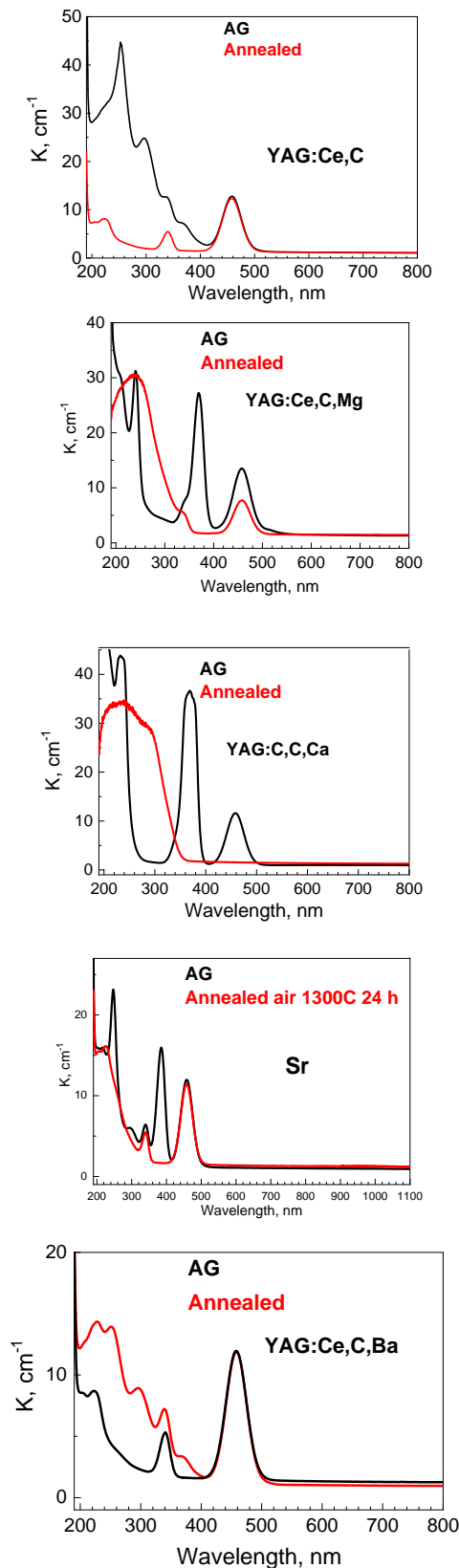


Fig. 2. Optical transmission spectra of YAG:Ce,C,A²⁺ before/after the annealing.

In Mg²⁺- and Ca²⁺-containing crystals, the absorption bands at 340, 458 nm related to 4f-5d transitions in Ce³⁺ weaken (Mg²⁺) or disappear (Ca²⁺) after the annealing with a

simultaneous increase in UV-absorption below 350 nm attributed to Ce⁴⁺-O²⁻ charge transfer complexes (Fig. 2). It is interesting that there are no any manifestations of Ce³⁺ in YAG:Ce,C,Ca²⁺ indicating that the quantity of cerium in the trivalent state is negligibly small. Previously we observed this phenomenon in YAG:Ce,Mg single crystalline fibers obtained by the micro-PD method. At the same time, the annealing does not affect the intensity of the Ce³⁺ bands in Sr²⁺- and Ba²⁺-codoped samples. Presuming the 458 nm peak intensity as a measure of Ce³⁺ concentration and no Ce⁴⁺ in crystals grown under the reducing conditions, a negligible concentration of Ce³⁺ remains in YAG:Ce,Ca, approximately half of Ce³⁺ remains in YAG:Ce,C,Mg²⁺, and no valency change is observed in Ba²⁺- and Sr²⁺-codoped YAG:Ce,C.

Meanwhile, the sharp absorption bands around 370 and 230 nm attributed to F⁺-centers in garnets [17] are suppressed in the annealed YAG:Ce,C codoped with Ca²⁺, Mg²⁺ and Sr²⁺, whereas UV-absorption related to Ce⁴⁺-O²⁻ charge transfer complexes remarkably enhances in the annealed crystals except YAG:Ce,C,Sr²⁺. Summarizing, post-growth annealing suppresses color centers at Ca²⁺, Mg²⁺, or Sr²⁺-doping and, while Ca²⁺ and Mg²⁺-doping also promotes tetravalent Ce. The increase of color center-related absorption in UV is observed with Ba²⁺ doping. From the point of compensation of lacking positive charge in the as-grown samples doped with Ca and Mg, anionic vacancies may form (one O²⁻ vacancy for 2 Ca²⁺), which capture electrons and form F⁺-centers. Therefore, an intense band at 370 nm in as-grown crystals in this case serves as an indicator of incorporation of divalent codopants. Its intensity is small in case of Ba, because this cation probably does not enter the lattice in sufficient quantity. The different behavior of the Ba²⁺-codoped sample may be attributed to a very large ionic radius of Ba²⁺ leading to formation of some additional defects at Y³⁺/Ba²⁺ substitution.

The decay times of UV-excited curves (not shown) in all the codoped crystals are within the 56-63 ns, and the curves are well fitted by single-exponential function. No certain trend before/after annealing. Meanwhile, some interesting features are revealed in the UV-visible excitation and emission spectra. Let us illustrate them on the example of Mg²⁺-codoped crystals (Fig. 3). While the excitation and emission spectra in all the codoped crystals after the annealing are identical with the Ce³⁺ excitation peaks at 220, 340, 460 nm and emission peak at 458 nm, there are some specific features on the spectra of as-grown crystals. Along with the mentioned Ce³⁺-related peaks, there is a broad emission peaked around 750 nm with a complex excitation spectrum. The latter is excited in both Ce³⁺ excitation peaks, and the 370 nm peak associated with F⁺-centers [18] and 520 nm peak of an unknown nature. Similar emission was registered by us in YAG:C without codoping with divalent cations [10] and related to carbon-related defects. However, all these bands disappear after the annealing in air.

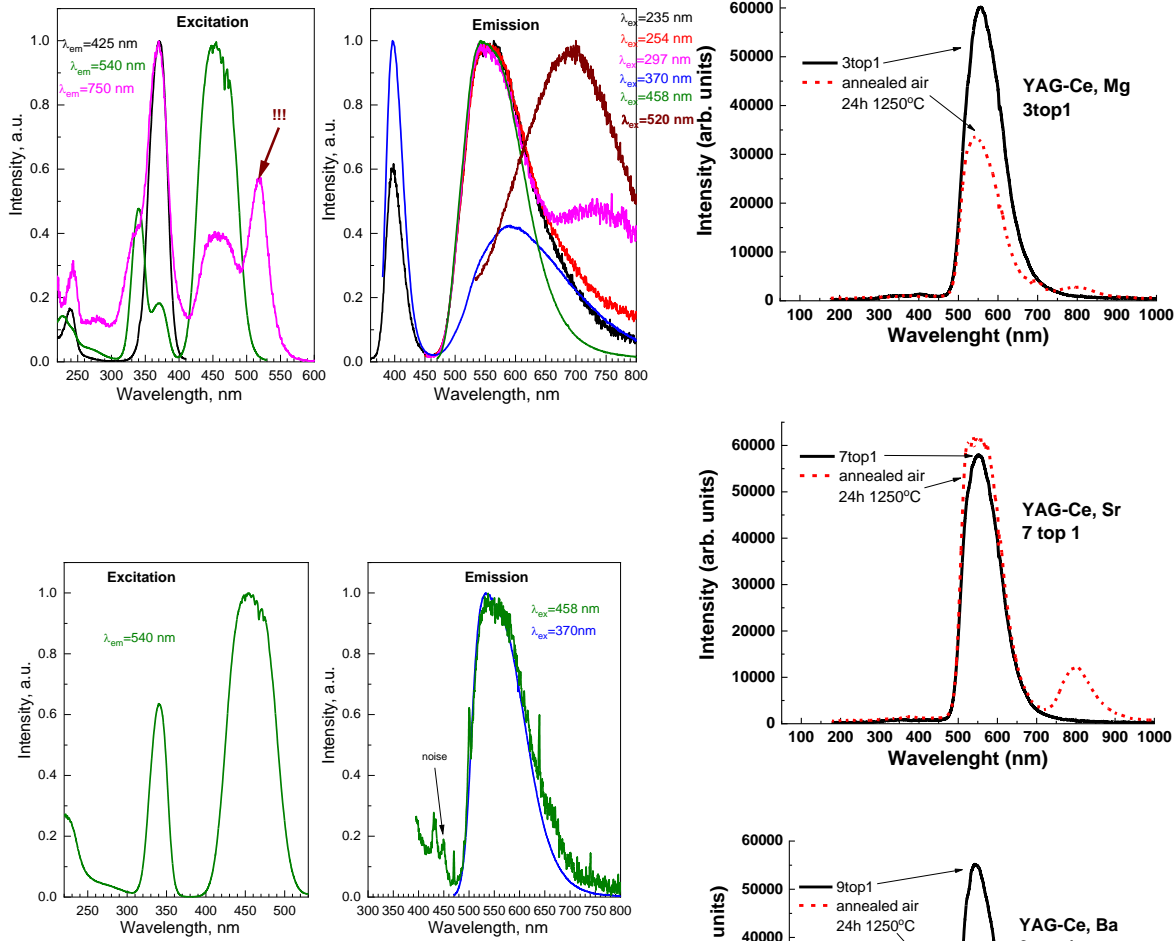


Fig. 3. Excitation and emission spectra of YAG:Ce,C,A²⁺ before (a) and after (b) the annealing.

B. Scintillation properties of YAG:Ce,C,A²⁺ crystals

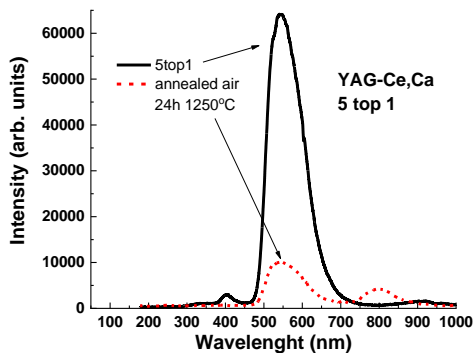


Fig. 4. Cathodoluminescence spectra of YAG:Ce,C,A²⁺ before/after the annealing.

Decrease in the Ce³⁺ peak intensity at 550 nm in the cathodoluminescence spectra (Fig. 4) after the annealing in all the samples except Sr²⁺-doped one, in general, corresponds to the weakening of Ce³⁺ absorption bands in Fig. 2, confirming the Ce transfer into the tetravalent state under the oxidizing conditions. Note the weakest intensity is registered in YAG:Ce,C,Ca where the Ce³⁺ absorption is negligible (see Fig. 2). Moreover, all the spectra contain a very weak band in UV-band related to color centers associated with antisite defects [17], and a wide band around 800 nm attributed to Fe³⁺ emission [19] rising after the annealing.

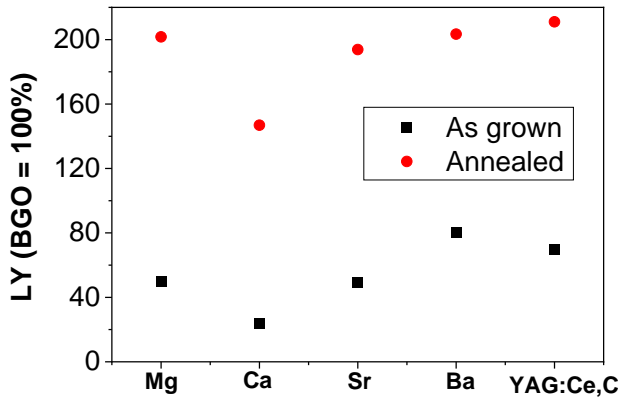


Fig. 5. Relative light yield (without the correction for PMT sensitivity) of YAG:Ce,C,A²⁺ before/after the annealing. 100 % corresponds to the BGO scintillator with a light yield of 8600 phot/MeV.

Switching to light yield, in Mg²⁺-, Sr²⁺-, Ba²⁺-doped crystals it is similar to that in YAG:Ce,C both before and after the annealing, while it is remarkably lower only in YAG:Ce,Ca²⁺ where a concentration of Ce³⁺ centres is very low (Fig. 5). This supports the suggestion by M. Nikl et al. regarding the positive impact of partial (but incomplete) transfer of Ce to the tetravalent state on scintillation performance [3]. It is interesting to note the sharp enhancement of light yield at simultaneous reducing the cathodoluminescence intensity after the annealing in all the samples, expect the Sr²⁺-codoped crystal where the cathodoluminescence peak intensity does not change remarkably.

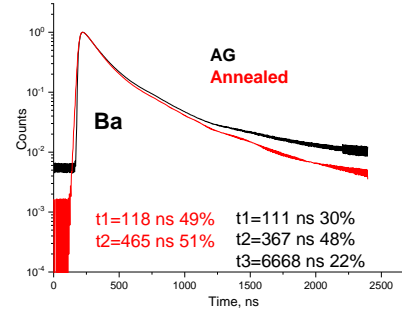
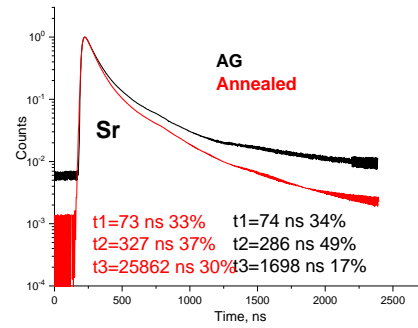
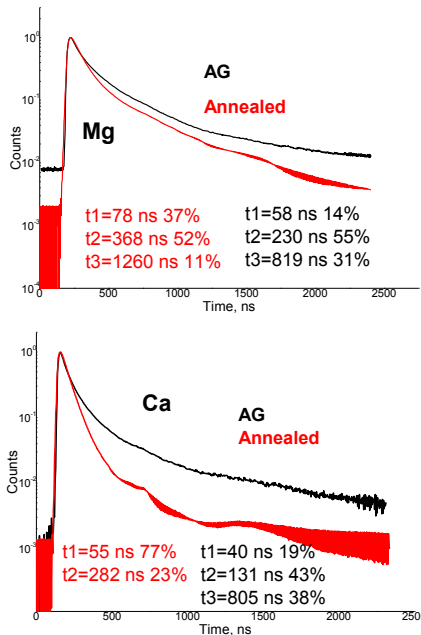


Fig. 6. Scintillation decay of YAG:Ce,C,A²⁺ before/after the annealing.

Scintillation decay accelerates after the annealing in all the samples (Fig. 6), most remarkably in YAG:Ce,Ca. The decay times at fitting with two- or three exponential functions are quantitatively compared in Table 1. The Ca²⁺- and Mg²⁺-codoping is the most promising for fast timing applications. However, the decrease in decay times in YAG:Ce,C,Ca is achieved at the expense of light yield, in agreement with the numerous data on C-free garnets [3-6]. As the main decay time of 55 ns in YAG:Ce,C,Mg²⁺ is too slow for the desired application, tuning the Ce³⁺ and codopant concentrations, or combined codoping with two divalent cations may be useful for further acceleration of scintillation decay.

TABLE I
SCINTILLATION DECAY TIMES (NS) AND CONTRIBUTIONS OF DECAY COMPONENTS (%) IN YAG:CE,C,A²⁺.

	As-grown			Annealed		
	τ_1	τ_2	τ_3	τ_1	τ_2	τ_3
Mg	58 (14%)	230 (55%)	819 (31%)	78 (37%)	368 (52%)	1260 (11%)
Ca	40 (19%)	131 (43%)	805 (38%)	55 (77%)	282 (23%)	
Sr	74 (34%)	286 (49%)	1698 (17%)	73 (33%)	327 (37%)	25862 (30%)
Ba	118 (49%)	465 (51%)		111 (30%)	367 (48%)	6668 (22%)
YAG: Ce,C				101 (19%)	359 (60%)	2260 (21%)



The main TSL peaks around 150 °C and 250-300 °C are observed in all as-grown crystals with different intensity ratios between them (Fig. 7). There is a common tendency of decreasing the TSL intensity after the thermal annealing, certifying a decrease in defect concentration and carrier trapping. The main remaining peaks are at 150, 200 and 310 °C (YAG:Ce,C,Mg), 220 and 325 °C (YAG:Ce,C,Ca), 150, 200, 310 °C (YAG:Ce,C,Sr), and 150, 200, 315 °C (YAG:Ce,Ba). The most striking difference between the samples is weakly manifested 150 °C peak, and the shift of >300 °C peak to higher temperatures in the Ca²⁺-codoped sample. In general, the structure of TSL curves is similar to that reported for oxygen-deficient YAG:C [20] and YAG:Ce in the form of single crystals and optical ceramics [21]. There is a tendency to a higher intensity of the high-temperature glow above 300 °C in the samples studied in the present work, which may point at deep traps associated with carbon-related defects; however, a detailed interpretation of the peak nature is complicated due to the large amount of possible defect types created at carbon-doping.

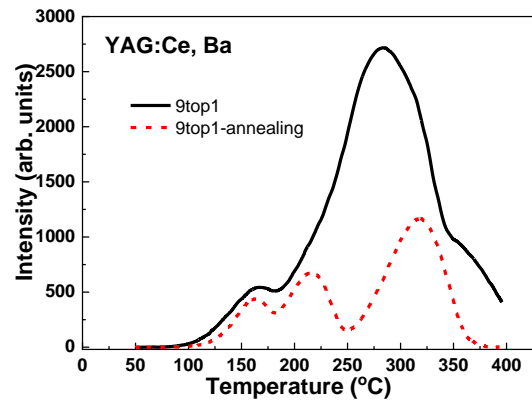
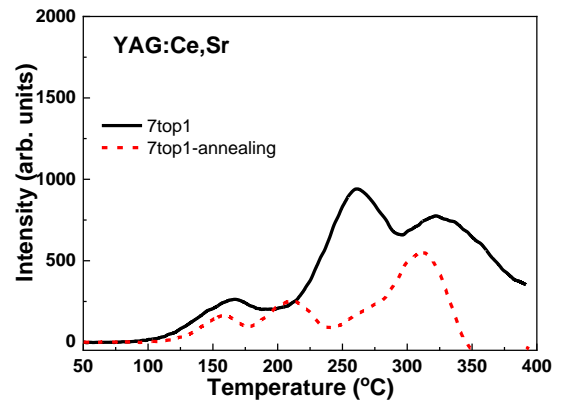
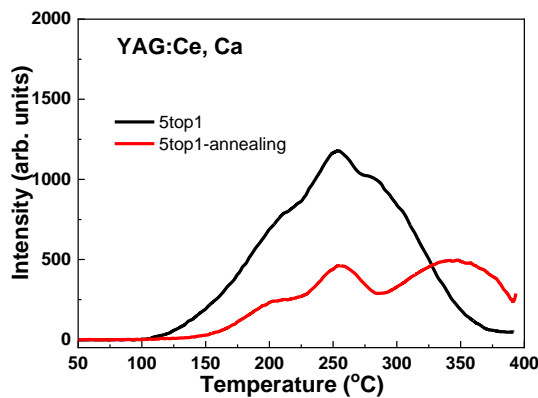
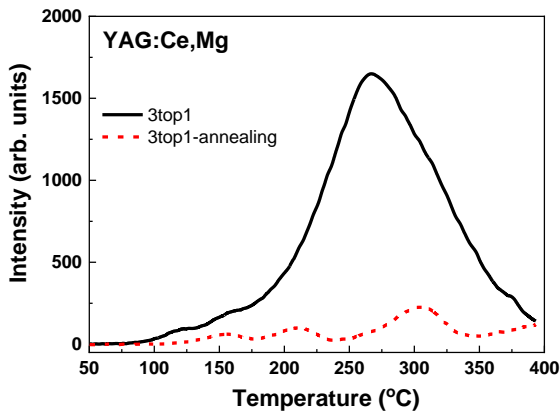
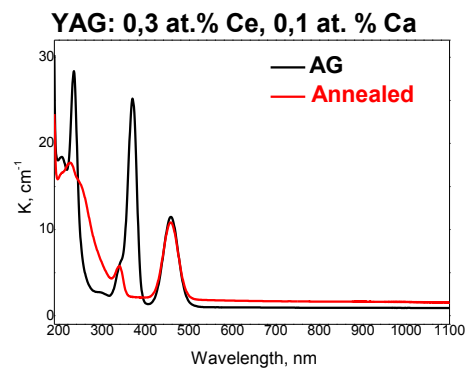


Fig. 7. TSL curves of YAG:Ce,C,A²⁺ before/after the annealing.

C. Tuning the codopant concentration in YAG:Ce,C,Ca²⁺.

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As the shortest decay times were registered in the Ca²⁺-codoped sample, the crystals with the codopant content from 0.1 to 1at.% and Ce content from 0.3 to 1at.% were grown thereby tuning the Ce/Ca ratio.



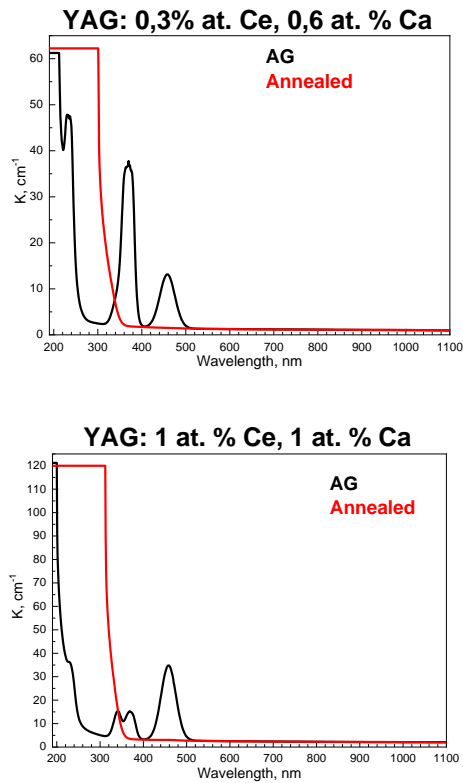


Fig. 7. Absorption spectra of YAG:Ce,C,Ca²⁺ crystals with different Ce/Ca ratios.

Expectedly, the Ce³⁺ absorption around 450 nm is completely suppressed after the annealing at the Ce/Ca ratios below unity, and the light yield monotonously decreases with Ca²⁺ concentration (Fig. 8). The main decay constant of around 55 ns has not decreased, but in the 1%Ce, 1%Ca sample its contribution reaches 96 %, with practically no slow components (Fig. 9, Table 2), and the light yield of approximately 17500 phot/MeV. Therefore, despite the remarkable decrease in light yield, the main decay time not falls below the 55-59 ns range corresponding to intrinsic radiation lifetime of Ce³⁺ in garnets [22]. The tradeoff between light yield and decay time was not resolved by the sole Ca²⁺ codoping, and the works on codoping are on the way.

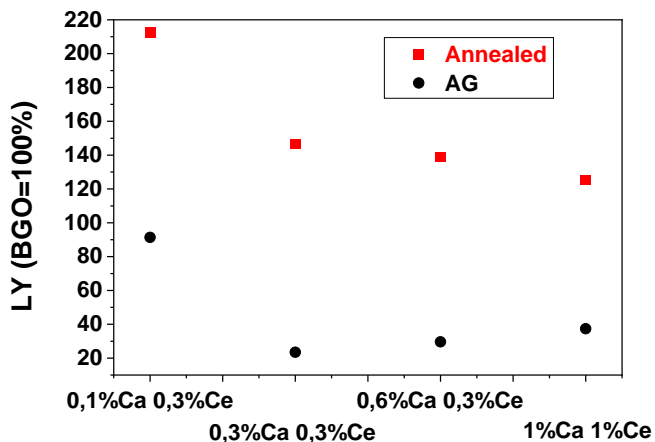


Fig. 8. Light yield of YAG:Ce,C,Ca²⁺ crystals with different

Ce/Ca ratios.

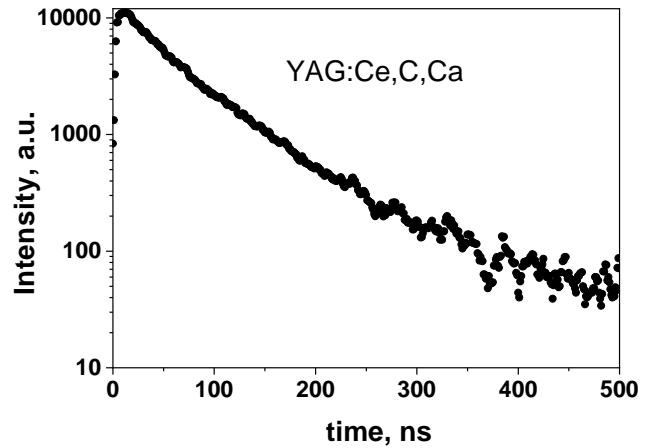


Fig. 9. Decay curve of YAG:1%Ce,C,1%Ca²⁺.

TABLE III
SCINTILLATION DECAY TIMES (NS) AND CONTRIBUTIONS OF DECAY COMPONENTS (%) IN YAG:CE,C,Ca²⁺ CRYSTALS WITH VARIED CE/CA²⁺ RATIO.

	As-grown			Annealed		
	τ_1	τ_2	τ_3	τ_1	τ_2	τ_3
0,1%Ca, 0,3%Ce	71 (22%)	275 (50%)	1345 (28%)	74 (52%)	362 (48%)	-
0,3%Ca, 0,3%Ce	40 (19%)	131 (43%)	805 (38%)	55 (77%)	282 (23%)	-
0,6%Ca, 0,3%Ce	46 (13%)	158 (47%)	992 (40%)	59 (75%)	270 (25%)	-
1%Ca, 1%Ce	69 (72%)	458 (28%)	-	55 (96%)	347 (4%)	-

IV. CONCLUSIONS

Effects of triple codoping of YAG:Ce,C with divalent cations was verified with Ba²⁺, Ca²⁺, Mg²⁺, Sr²⁺. Although the main decay time was not decreased below the Ce³⁺ intrinsic lifetime of ca. 55 ns in garnets, the contribution of long components of scintillation decay related to intrinsic lattice defects was reduced to 4% by tuning the Ce/Ca ratio while retaining the light yield of 17500 phot/MeV. Ca²⁺, Mg²⁺ and Sr²⁺ promoted the formation of tetravalent Ce and acceleration of scintillation decay by the mechanisms priorly described for Ce-doped garnets and orthosilicates. Ba²⁺ due to its large ionic radius poorly entered the lattice and barely affected the optical and scintillation performance. The trade-off between light yield and scintillation decay was not overcome by doping with the single cations, and further work should be focused on combined codoping with different divalent cations.

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