Drastic Scintillation Yield Enhancement of YAG:Ce with Carbon Doping

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Scintillation performances of carbon-doped YAG and YAG:Ce crystals obtained by the Czochralski method under novel conditions of Ar+CO reducing atmosphere is compared to their counterparts obtained by conventional technologies. While light yield and energy resolution in YAG crystals grown under different conditions is similar, a fast luminescence decay component of 4–6 ns is observed in YAG:C grown by the new procedure. Optimization of post-growth thermal annealing procedure of YAG:Ce,C scintillator provides the very high light yield of 28 200 phot MeV⁻¹, and the energy resolution of 7.8–8.5% at 662 KeV.

1. Introduction

New high-energy physics experiments at colliders (see, for ex., Coupard et al.[1]), as well as future generation of medical tomography equipment[2] need bright scintillators with faster luminescence decay compared to currently widely used Ce³⁺-doped scintillators. Y₃Al₅O₁₂ (YAG) garnet crystals are considered as an efficient host for laser, phosphor, and scintillation materials. Scintillation properties of undoped YAG were barely explored in detail due to low light yield and slow luminescence decay. Meanwhile, Ce-doped garnets did not attract much attention until the development of extremely slow luminescence decay compared to currently widely used Ce³⁺-doped garnets, such as YAG:Ce, without considering solid solutions with other cations. This is important, because Gd₃Al₅-xGaₓO₁₂:Ce (GAGG:Ce) and related Ga-containing crystals have to be grown in inert gases enriched with oxygen to reduce Ga evaporation. Only Ir crucibles are capable to withstand such atmosphere at temperatures near 2000 °C, if oxygen content does not exceed 1–2 vol.%. Nevertheless, Ga evaporation from the melt under these conditions is still rather strong. This, evidently, causes the tendency of crystals to cracking. As a result, the use of Ir crucibles and expensive thermal insulation material, as well as the high energy consumption during the crystal growth of rare-earth garnets, makes the crystals expensive for some applications. The economic factor is a problem also for other high-melting point complex oxide scintillators, such as Lu₂SiO₅:Ce (LSO:Ce) and Lu₁₂₋ₓYₓSiO₅:Ce (LYSO:Ce). All these factors promote the development of low-cost crystal growth technologies.

Procedures for aluminum garnets and perovskites growth in Mo and W crucibles have been developed since 1970–80s[7–9] until much more recently.[10–12] Meanwhile, a new procedure of growth[13,14] and post-growth heat-treatment[15] for garnet-based crystals was developed by some of authors of this work, and a series of undoped and Ce-doped YAG crystals were produced. Among the basic features of the novel procedure there is the use of cheap graphite heat insulation in crystallizer and reducing Ar+CO growth atmosphere. The latter is a cause of few hundreds wt. ppm (up to 1 at%) carbon incorporation in crystals.[15] As carbon impacts on YAG optical properties,[15] the crystals obtained in these conditions were denoted as carbon-doped crystals (YAG:C and YAG:Ce,C).

In this paper the scintillation parameters of YAG:C and YAG:Ce,C crystals grown by the developed procedure are compared with YAG and YAG:Ce grown by the conventional technologies.
2. Methods

2.1. Crystal Growth

YAG:C and YAG:Ce,C crystals were grown by the developed procedure in Mo and W crucibles using graphite heat insulation under Ar+CO reducing atmosphere. The growth procedure details are described elsewhere.\cite{13,14} The carbon concentration was up to 1 at% in YAG:C crystals. The Ce concentration in YAG:Ce,C was around 0.1 at%. Carbon concentration was not measured in YAG:Ce,C, but it is presumed to be the same as in YAG:C, because the crystal growth procedure was the same. Cerium was introduced in the form of CeAlO$_3$ single crystals, whose production technology was developed by us in Fujimoto et al.\cite{16} As-grown YAG:C were greenish, and YAG:Ce,C were yellow-greenish as the result of interaction with the reducing atmosphere (Figure 1). The post-growth heat treatment of crystals at 1200 °C under air and at 1500–1850 °C under Ar+CO atmosphere provided the bleaching of YAG:C and less deep yellow coloration of YAG:Ce,C. The reference YAG and YAG:Ce crystals were grown by the Czochralski method from Ir crucibles in Ar gas atmosphere. Samples with the dimensions of 10×10×2 mm$^3$ were fabricated for optical and scintillation measurements.

2.2. Measurement of Spectral Properties

The absorption spectra were measured using a Specord 40 spectrophotometer (Analytik Jena AG). X-ray luminescence (RL) spectra were measured using a home-made apparatus featuring a CCD detector (Jobin-Yvon Spectrum One 3000) coupled to a monochromator (Jobin-Yvon Triax 180). RL irradiation was performed by a Philips 2274 X-ray tube.

2.3. Determination of Light Yield and Energy Resolution

Light yield (LY) and energy resolution (R) were measured under irradiation with a 662 keV $^{137}$Cs gamma source. A R1307 Hamamatsu PMT ran at 800 V and HV with linear dynode voltage divider were used for registration of YAG:Ce,C parameters. Meanwhile, a R2059 Hamamatsu PMT run at 1400 V with better sensitivity in UV-range was used for measurements with YAG:C. PMT output was connected to the charge-sensitive preamplifier BUS 2–94 and BUI-3K shaping amplifier. Signal from preamplifier was shaped by a shaping amplifier with 2 μs (for YAG:C) and 8 μs (for Ce-doped YAG) shaping times. In order to collect the whole scintillation light the samples together with open part of PMT photocathode were covered with Teflon reflector. No optical contact between samples and PMT window was provided. For absolute light yield determination the yields were compared to Bi$_2$Ge$_2$O$_12$ (BGO) produced at ISMA, and Lu$_2$SiO$_5$ (LSO:Ce) sample obtained from Proteus Inc., USA. The quantum efficiencies were 0.1867 (BGO), 0.1158 (YAG:Ce,C), 0.2578 (LSO:Ce) for measurements with R1307 PMT, and 0.1472 (BGO) and 0.2108 (YAG:C) for measurements with R2059 PMT based on the luminescence spectra of the crystals and the PMT quantum efficiency. Energy resolutions of $^{137}$Cs 662 keV peaks are determined by approximation of the obtained pulse-height spectra with Gaussian function.

2.4. Determination of Decay Times

Scintillation decay times were determined at X-ray excitation (50 kHz excitation repetition rate, FLE 400 filter $\lambda > 400$ nm). Pulsed X-ray decay measurements were performed using a Hamamatsu N5084 light-excited X-ray tube set at 30 kV as irradiation source. The optical excitation of the tube was performed with a Hamamatsu PLF-10 picosecond light pulser. The scintillation decay was registered by a PMA 165-C PMT from PicoQuant operating in single photon counting regime.
According to the data sheet, the transit time is below 180 ps and the rise and fall time are 750 ps providing a time resolution of 180 ps.

3. Experimental Results and Discussion

3.1. YAG:C

The as-grown crystals are colored, while the annealed crystals are highly transparent both in UV- and in the visible. In annealed YAG:C the color centers are not re-formed under any subsequent reducing or oxidizing annealing (see Tkachenko et al.\[15\] for details). The irreversible bleaching was observed for YAG:C crystals grown in W, Mo, and Ir crucibles (if raw material was prepared under Ar$^+$CO atmosphere). This means that the possible introduction of crucible material atoms in the crystals is not responsible for this phenomenon. The lack of coloration after subsequent annealing was attributed单纯的 centers of electron capture linked to complex C-related defects. Thus, a high transparency of annealed YAG:C crystals and their scintillation parameters sustain under any further annealing.

Light yield (10000–14000 phot MeV$^{-1}$), energy resolution (17% at 662 keV) and scintillation decay ($\tau = 400$ ns + slow component) of annealed crystals are similar to those reported in Seki et al.\[11\], Fujimoto et al.\[16\] and Varney\[17\] while light yield and energy resolution of as-grown YAG:C are significantly worse due to the formation of color centers (Table 1).

Meanwhile, colored YAG:C, apart of few hundred ns component ascribed to the self-trapped exciton (STE) emission, and long microsecond components, possesses a very fast luminescence decay component with $\tau = 4–6$ ns under X-ray excitation. The decay curves are well fit by the three-exponential decay. The contribution $W_1$ of the fast component with $\tau_1 = 4.8$ ns at fitting (Figure 2) to the light sum within the 30 microsecond gate is $\approx 2\%$, according to the equation (1):

$$W_1 = \frac{\int t_1 A_1}{\int t_1 A_1 + \int t_2 A_2 + \int t_3 A_3} \times 100. \quad (1)$$

The fast nanosecond component under selective UV-excitation and synchrotron excitation was already reported before in the literature. Probably, the most detailed analysis of emission centers in undoped garnets was presented in Zorenko et al.\[18\] where the 2–3 ns decay components with emission band peaked around 400 nm (3.095 eV) and excitation at 3.33; 5.37; 6.56; 7.3 eV were attributed to F$^+$-centers localized near Y$_{Al}$ antisite defects. The same luminescence band peaked at 397 nm and excited at 370 and 236 nm was registered in our YAG:C crystals\[19\] (in that work they are denoted as "Type III" crystals). Similar data on fast (<10 ns) luminescence decay in garnets by $\gamma$-ray excitation were reported previously in Varney\[17\] but it is impossible to evaluate its contribution based on the there presented data.

Table 1. Scintillation parameters of YAG:C and YAG crystals.

|                      | Colored (as grown) YAG:C (this work) | Colored YAG | Transparent (annealed) YAG:C (this work) | Transparent YAG |
|----------------------|--------------------------------------|-------------|------------------------------------------|-----------------|
| Light yield, phot/MeV | <10 000                              | 50 000/5.5 MeV\[11\] | up to 14 000                             | 14 300\[16\] |
| Luminescence decay times, ns | $\approx 5 +$ slow                | 3-4 + slow\[17\] | 400 + slow\[17\]                         | $\approx 460$\[16\] |
| Energy resolution at 662 keV, % | Not identified                    | 16–18\[17\] | 17                                       | 11–14\[17\] |

Figure 2. Scintillation decay after X-ray irradiation of colored YAG:C crystal fit by the function $y = 0.003 + 0.883 \times \exp(-t/4.8) + 0.064 \times \exp(-t/296.4) + 0.058 \times \exp(-t/3137.9);$ inset: the same data within the time range of first 60 ns.

Figure 3. Typical X-ray luminescence spectra of YAG and YAG:C crystals.
Figure 3. X-ray excited luminescence spectra are shown in Figure 3. While YAG spectrum demonstrates just one dominant band peaked at 310 nm, which is evidently a superposition of emission of excitons trapped near antisite defects and Gd\(^{3+}\) impurity emission, the spectrum of transparent YAG-C crystals contains much wider luminescence band in the same range with the main peak near 300 nm of AD-related exciton emission, and shoulder at \(\approx\)245 nm attributed to self-trapped exciton emission. The wide 300 nm peaked band is a superposition of peaks of exciton emission related to different types of AD-related defects. Unlike transparent crystals, in colored YAG-C the complex luminescence band arises at \(>\)395 nm, which is evidently related to the registered fast \(\mathrm{F}^+\) center emission. Similar luminescence band was observed in YAG grown under \(\mathrm{Ar}+\mathrm{H}_2\) atmosphere, and the persistent blue luminescence was revealed in carbon-doped YAG, which was proposed for dosimetry applications. However, we have not noticed a clear correlation between the contribution of the fast luminescence decay component and intensity of the \(>\)370 nm luminescence bands on X-ray luminescence spectra.

3.2. YAG:Ce,C

While the light yield of as-grown YAG:Ce,C is similar to the light yields of YAG:Ce reported in most of the papers, the light yield of YAG:Ce,C after thermal annealing under air or \(\mathrm{Ar}+\mathrm{CO}\) atmosphere increases by 1.5–2.0 times compared to the as-grown crystal (Figure 4. Table 2). The highest light yield registered after annealing in air is 330% relatively to BGO taking into consideration the quantum efficiency of these scintillators with a R1307 PMT. Assuming the averaged light yield of BGO reference sample of 8600 phot MeV\(^{-1}\) according to 8000–9000 phot MeV\(^{-1}\) values prevailing in literatures, Moszynski et al., Salacka et al., Sakai, and Holl et al., the light yield of air-annealed YAG:Ce,C is 28 200 phot MeV\(^{-1}\) (Table 2). The LSO:Ce reference light yield of 28 000 phot MeV\(^{-1}\) determined by our procedure lies within the standard values for this scintillator, see for example, Conti et al., and confirms the correctness of our data. To the best of our knowledge, just one paper reports the similar light yield around 30 000 phot MeV\(^{-1}\) in YAG:Ce,C, though without the pulse height spectrum (Table 2). The energy resolution in air-annealed sample presented in Figure 4 is 8.1%, though it ranges from 7.8 to 8.5% in different samples.

The growth and post-growth annealing under the reducing conditions should promote the reduction of cerium into the optically active trivalent state. We presume that in as-grown YAG:Ce,C a high Ce\(^{3+}\) concentration does not lead to light yield increase because of high concentration of defects in crystals. The light yield improvement after the reducing annealing coincides with the suppression of the absorption band peaked at 371 nm and related to \(\mathrm{F}^+\) centers, as shown in Figure 5. As one might expect the inverse effect of increase of trap and color center concentrations at reducing annealing, we attribute the absorption decrease, by analogy to YAG:C, to the presence of carbon, which creates carrier capture centers competing for electrons with \(\mathrm{F}\) and \(\mathrm{F}^+\) centers. Meanwhile, the energy resolution in YAG:Ce,C deteriorates after the reducing annealing.

The air annealing significantly improves both light yield and energy resolution. This is evidently attributed to the elimination of oxygen vacancies and related defects. According to the intensity of Ce\(^{3+}\) absorption bands at 458 and 340 nm, and absorption band around 300 nm attributed to Ce\(^{4+}\)–O\(^2-\) charge transfer complexes, the weakening of Ce\(^{3+}\) bands and reinforcement of Ce\(^{3+}\)–O\(^2-\) band in the air-annealed sample compared to CO-annealed sample (see the difference spectrum at the inset in Figure 5) certify a partial oxidation of Ce\(^{3+}\) into Ce\(^{4+}\). According to Nikl et al., Ce\(^{4+}\) competes for electron

| Crystal     | Growth method | Crucible material | Light yield [phot MeV\(^{-1}\)] | Energy resolution [%] | Scintillation decay time [ns] | Background level [%] after 3 μs |
|-------------|---------------|-------------------|---------------------------------|------------------------|-----------------------------|-----------------------------|
| YAG:Ce,C    | Cz\(^{3+}\)   | Mo/W              | 14 200                          | 9.7                    | 73 (30%), 254 (70%)         | 2.94                        |
| YAG:Ce,C    | Cz\(^{3+}\)   | CO-anneal.        | 21 700                          | 12.9                   | 100 (77%), 331 (23%)        | 1.77                        |
| YAG:Ce,C    | Cz\(^{3+}\)   | Air-anneal.       | 28 200                          | 7.8–8.5                | 98 (79%), 349 (21%)         | 1.30                        |
| YAG:Ce[C\(^{4+}\)] | HDC\(^{3+}\) | Mo                | 15 000–18 000                   | 8–10                   | No data                    | No data                     |
| YAG:Ce[C\(^{4+}\)] | Cz\(^{3+}\)   | No data           | 16 700                          | No data                | No data                    | No data                     |
| YAG:Ce[C\(^{4+}\)] | Cz\(^{3+}\)   | No data           | No data                         | No data                | No data                    | No data                     |
| YAG:Ce[C\(^{4+}\)] | Cz\(^{3+}\)   | Air-anneal.       | 30 250                          | No data                | No data                    | No data                     |

a) Czochralski method; b) Horizontal directional freezing method.
capture with shallow electron traps in garnets, and the coexistence of Ce\(^{3+}\) and Ce\(^{4+}\) reduces electron trapping and promotes fast and bright scintillation.

Comparison of X-ray luminescence spectra (Figure 6) indicates that both position and shape of Ce\(^{3+}\) luminescence bands at 530 nm are the same in YAG:Ce and YAG:Ce,C, and they are not affected by the thermal treatments of YAG:Ce,C. Note that the most intense X-ray excited Ce\(^{3+}\) luminescence is registered in CO-annealed samples, while air-annealed YAG:Ce,C possesses the highest light yield. The 310 nm complex band in YAG:Ce, by the analogy to YAG, is related to Y\(_{Al}\) antisite luminescence and Gd\(^{3+}\) emission. The excitation peak at 273 nm and emission peak at 312 nm clearly related to Gd\(^{3+}\) luminescence were detected in YAG:Ce spectra (not shown in this paper). Meanwhile, the weakening of the band peaked 310 nm suggests the decrease of concentration of antisites in Ce,C-codoped crystals. The absence of Gd\(^{3+}\)-related peak in carbon-codoped samples can be attributed to the enhanced energy transfer from Gd\(^{3+}\) to Ce\(^{3+}\). Therefore, the improved energy transfer to Ce\(^{3+}\) luminescence centers is an additional reason of the light yield improvement in YAG:Ce,C scintillator.

Pulsed X-ray luminescence decay curves of YAG:Ce,C within the 2\(\mu\)s range were fit by the double exponential function 
\[ y = y_0 + A_1 \times \exp(-t/\tau_1) + A_2 \times \exp(-t/\tau_2) \]
The scintillation decay constants of as-grown YAG:Ce,C (Table 2) are similar to those published in literature (see, for ex., Mihokova et al.\(^{[24]}\)) with the fast (\(\tau_1 = 73\) ns) and slow (\(\tau_2 = 254\) ns) components. Although the calculated decay constants are somewhat longer in the case of the annealed samples (98, and 100 ns), the overall decay is faster, as one may see in Figure 7, due to the decrease in carrier trapping responsible for the delayed luminescence. The phosphorescence level (background) after 3\(\mu\)s decreases by more than twice after the thermal annealing (see Table 2).

Despite the very high measured light yield, we suppose that there is still room for further improvement of scintillation parameters by the optimization of Ce concentration and other parameters of the preparation process.

4. Conclusions

Scintillation performance of carbon-doped YAG and YAG:Ce crystals is explored. The YAG:C parameters are similar to those in YAG obtained by other methods. Meanwhile the fast luminescence with \(\tau \approx 5\) ns attributed to F\(^{+}\)-centers under extrinsic excitation was revealed in as-grown YAG:C.

High-quality YAG:Ce,C crystals were obtained by the proposed Czochralski process using Mo/W crucibles and optimized post-growth annealing with a very high light yield of up to 28 200 phot MeV\(^{-1}\) and a good energy resolution of % at 662 keV. The contribution of fast \(\approx 100\) ns luminescence decay component in air-annealed YAG:Ce,C reaches 79%.

The obtained scintillation parameters are the result of complex interactions between electrons and holes with cerium in different valence states, carbon, as well as various types of intrinsic defects in the garnet structure. Thus, scintillation mechanisms in YAG:Ce,C still need further detailed study.
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Conflict of Interest

The authors declare no conflict of interest.

Keywords

light yield, scintillators, YAG, YAG:Ce

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