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FREE CARRIERS DYNAMICS IN HEAVY SCINTILLATION MATERIALS WITH GARNET STRUCTURE

Abstract. Ce-doped $Lu_3Al_5O_{12}$ (LuAG), and $Gd_3Al_2Ga_3O_{12}$ (GAGG) crystals with and without codoping by Mg^{2+} ions have been studied by the nonlinear absorption spectroscopy method. A faster rise time of transient optical absorption has been observed in all crystals codoped with Mg in comparison to Mg-free samples. A significant difference in the time evolution of the differential optical density in GAGG in comparison to LuAG crystals is revealed. In gadolinium garnets an absorption band peaked in the blue-green range and decaying with characteristic time of ~2 ps is observed. This band is considered to be due to absorption of free electrons before their trapping by Ce^{3+} doping ions. A broad transient absorption band in the yellow-red region is attributed to absorption from the Ce^{3+} excited states.

Keywords: transient absorption, scintillators, luminescence rise, luminescence kinetics, garnets

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ДИНАМИКА СВОБОДНЫХ НОСИТЕЛЕЙ В ТЯЖЕЛЫХ СКИНТИЛЯЦИОННЫХ МАТЕРИАЛАХ СО СТРУКТУРОЙ ГРАНАТА

Аннотация. Легированные ионами Ce кристаллы $Lu_3Al_5O_{12}$ (LuAG) и $Gd_3Al_2Ga_3O_{12}$ (GAGG) с и без кодопирования ионами Mg^{2+} исследованы методом нелинейной абсорбционной спектроскопии. Ускорение времени нарастания переходного оптического поглощения наблюдалось в кристаллах, кодопированных ионами Mg, по сравнению с образцами без Mg. Выявлено существенное различие во временной эволюции дифференциальной оптической плотности в GAGG в сравнении с кристаллом LuAG. В гадолиниевых гранатах наблюдается полоса поглощения в сине-зеленом диапазоне с затуханием с характерным временем ~2 пс. Эта полоса соотнесена поглощению свободными электронами до их захвата примесными ионами Ce^{3+}. Широкая полоса переходного поглощения в желто-красной области обусловлена поглощением из возбужденных состояний ионов Ce^{3+}.

Ключевые слова: переходное поглощение, сцинтилляторы, разгорание люминесценции, кинетика люминесценции, гранаты

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Introduction. The free carriers dynamics plays a crucial role in forming of the scintillator response and Coincidence Time Resolution (CTR) measurements. The achievable time CTR depends on the interplay between the light yield, light collection inside the scintillation element and the kinetics of the scintillation response [1]. Recently, Ce-doped garnets with the chemical formula $M_3A_5O_{12}$, in which the metal M can be either a single yttrium / rare earth ion or their mixture; and the metal A can be Al, Ga

or their mixture, have been extensively studied. They have a high light yield, the scintillation decay time shorter than 100 ns, and their emission peaks in the range between 520–560 nm, which perfectly matches the spectral sensitivity spectrum of silicon photo-multipliers (SiPM).

The codoping of Ce-doped scintillation materials with aliovalent ions has been recently demonstrated to be a promising approach to improve the scintillation properties, especially in the scintillators with a garnet-type crystal matrix [2, 3]. Such improvement is a combined result of the modification of electronic excitation transfer processes [4, 5].

Moreover, it causes a significant acceleration of the luminescence rise and decay times, as observed in $Gd_3Al_2Ga_3O_{12}$:Ce (GAGG:Ce) crystals codoped with Mg [6]. This leads to an improvement of the coincidence time resolution, as recently demonstrated with GAGG:Ce and $Lu_3Al_5O_{12}$:Ce (LuAG:Ce) crystals [7, 8].

In comparison with LuAG crystal, GAGG has a particular feature: it contains numerous levels of ^7f electronic states of Gd^{3+} ions in the band gap. As recently shown [9], the ground state ^8S of Gd^{3+} ions in GAGG crystals is located inside the valence band, about 1 eV below its top. Thus, a substantial difference in the excitation transfer to the radiating level of Ce^{3+} in GAGG is expected with respect to Gd-free garnets.

Since the luminescence build-up is caused by the population of the Ce^{3+} radiating levels, studying the time evolution of the population is a powerful tool to investigate both the luminescence response and the dynamics of free carriers before their capture by trapping and radiative centers.

To study these processes, the population of the lowest excited levels can be probed by exploiting the optical pump and probe technique: a femtosecond pump pulse is used for photo-excitation, while the probe pulse of similar duration but with a variable delay with respect to the excitation pulse is used to probe the free carrier density and the population of the excited electronic energy levels. In the current paper, a comparison of the fast excitation transfer processes in Ce-doped GAGG and LuAG crystals, with and without Mg-codoping, is discussed.

Experimental methods. Four Ce-doped garnet samples used in this study were grown by Czochralski method. The standard scintillation and optical properties of these samples were measured and discussed in [7].

The study of non-equilibrium carrier dynamics was performed using a pump-probe spectrometer based on an original femtosecond $Ti:Al_2O_3$ pulsed oscillator and a regenerative amplifier, both operating at 10 Hz repetition rate. The laser pulse duration and energy after amplification were 140 fs and up to 0.5 mJ, respectively, while the wavelength was tunable over the spectral range from 770 to 820 nm. The pulses of the fundamental frequency (the wavelength λ_{max} was set at 790 nm for the present study) were divided after the output of the amplifier into two parts at a ratio of 1:4. The beam of higher intensity was converted to the third harmonic ($\lambda \approx 263$ nm, pulse energy E up to 12 μJ) and used as the pump pulse. The pulse energy was selected low enough to prevent the sample surface from optical damage by the laser pulse. The second beam of smaller intensity was used as a probe. After passing the delay line, the probe pulse was converted to a white supercontinuum generated by focusing the beam into a 1 cm long water cell. By using a semi-transparent mirror, the supercontinuum radiation (360–800 nm) was subdivided into two pulses (reference and signal) of similar intensity. Both pulses were focused on the sample by mirror optics. The reference pulse was exploited to eliminate the impact of shot-to-shot instability of supercontinuum. It passes the sample always before the pump pulse. The pump-induced change of the optical density was calculated as:

$$\Delta D(\lambda, \Delta t) = \lg[(E_{sg}(\lambda) / E_{ref}(\lambda))/(E^*_{sg}(\lambda, \Delta t) / E_{ref}(\lambda, \Delta t))],$$

where E_{sg}, E^*_{sg}, and E_{ref} are the energies of the signal pulses passed the sample before and after the pump pulse, and the reference pulse, respectively. The beam spot diameter was 0.5 mm. The spectra of both pulses were recorded in the spectral range from 400 to 700 nm for each laser shot by a system including a polychromator equipped with a CCD camera and digitally processed. All measurements have been performed at room temperature.

Results. The absorption spectra of the crystals under study, measured through 2 mm thickness using a Perkin Elmer (Lambda 650 UV/VIS) spectrometer, are presented in Fig. 1. All the Mg-free crystals exhibit the 4f–5d absorption bands corresponding to inter-configuration transitions of Ce^{3+} ions. The samples

a   *b*
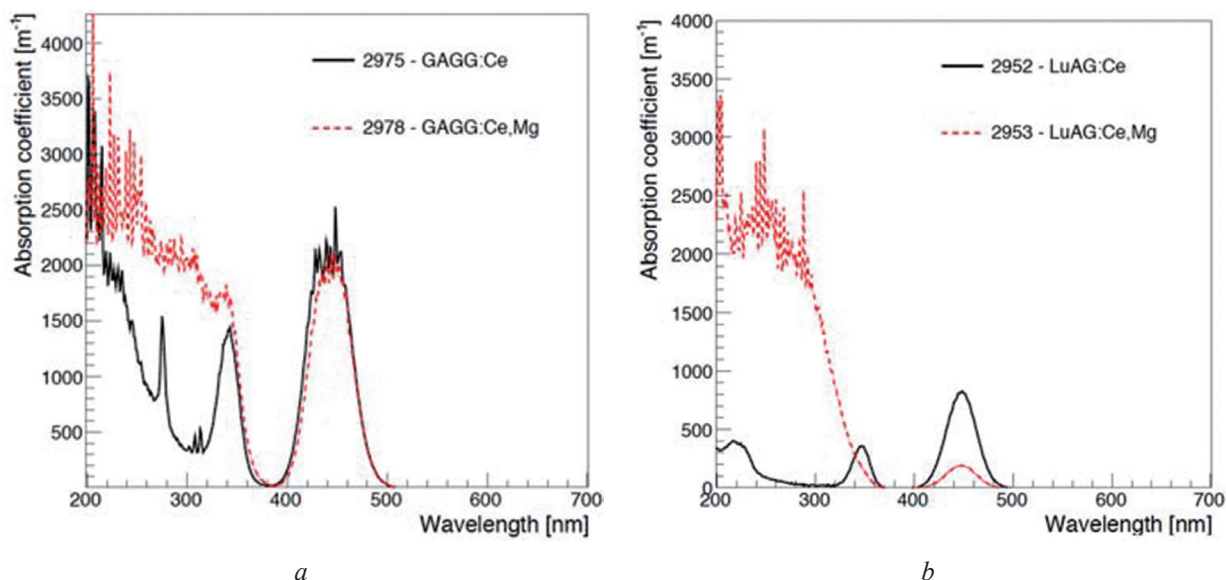
Fig. 1. Absorption spectra of GAGG:Ce without (black solid) and with Mg codoping (red dotted) (*a*);
of LuAG:Ce without (black solid) and with Mg codoping (red dotted) (*b*)

codoped with Mg^{2+} ions additionally show a broad absorption band in the UV region (below 300 nm). This band, related to the charge transfer (CT) transition from the valence band to a defect stabilized by Mg^{2+} (e.g., Ce^{4+} ions), is more pronounced in the LuAG:Ce crystals having the lowest concentration of Ce^{3+} ions. The absorption spectrum of the GAGG:Ce sample also shows a set of narrow absorption bands in the UV range corresponding to the transitions from ^8S ground state to the numerous components of P, I, and D terms split by spin–orbit interaction.

The transient induced absorption spectra of all samples for different delay times between the pump and the probe pulses are shown in Fig. 2–5. The pump wavelength of 263 nm was selected for excitation to avoid direct excitation of Gd^{3+} states in GAGG crystals. For all the samples under study, this wavelength excites the Ce^{3+} ions in the third component of the inter-configuration transition $4f^1$–$5d^14f^0$. Since the corresponding excited level is located in the conduction band (CB) the delocalization of the non-equilibrium electrons and their capture might easily occur. In addition, the pump causes CT transitions in the defect centers due to the divalent ion introduced by the Mg-codoping. The differential optical density spectra of GAGG:Ce show a drop around 440–450 nm, in correspondence with the first $4f^1$–$5d^14f^0$ allowed transition of Ce^{3+} ions. This occurs due to a relatively larger concentration of Ce^{3+} in this sample with respect to the lower dopant concentration in LuAG.

a   *b*
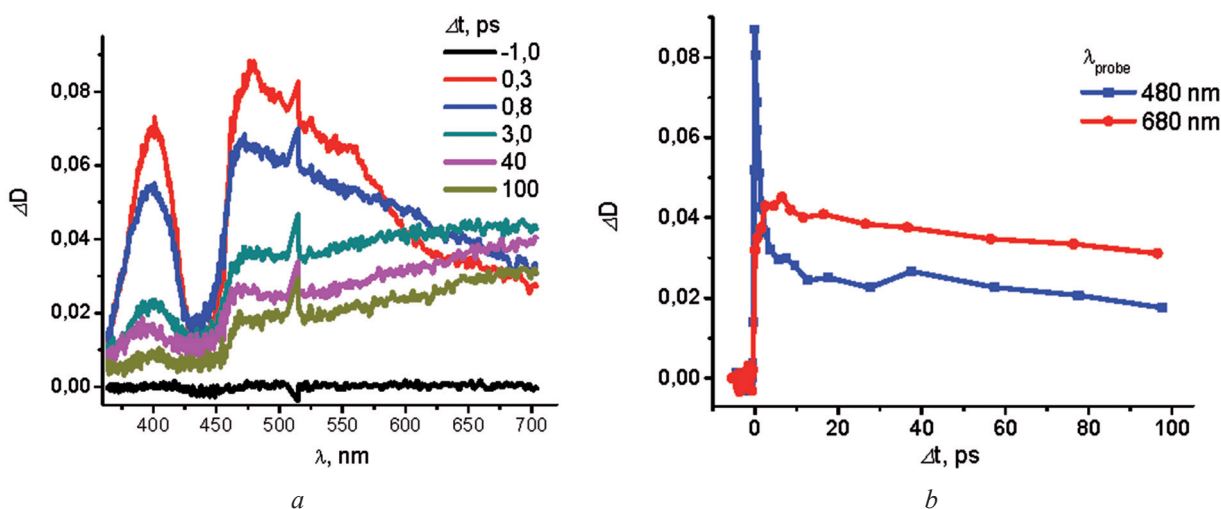
Fig. 2. Transient induced absorption spectra of GAGG:Ce single crystal at different time delays
between the pump and probe pulses (indicated) at pump pulse energy of 10 μJ (*a*);
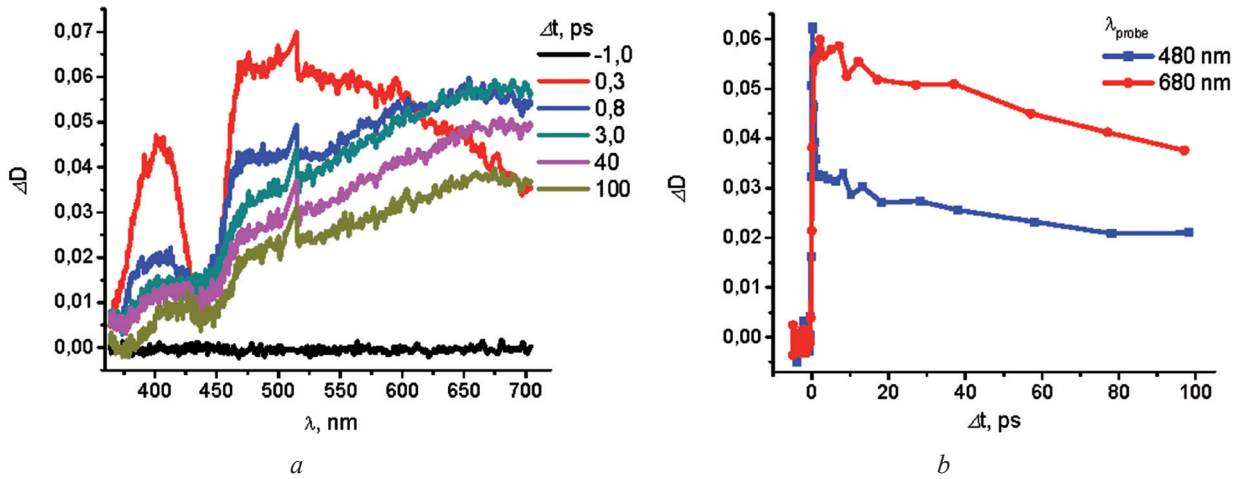and kinetics of probe beam absorption at the wavelengths of 480 and 680 nm (*b*)

Fig. 3. Transient induced absorption spectra of GAGG:Ce,Mg single crystal at different time delays
between the pump and probe pulses (indicated) at pump pulse energy of 10 µJ (*a*);
and kinetics of probe beam absorption at the wavelengths of 480 and 680 nm (*b*)
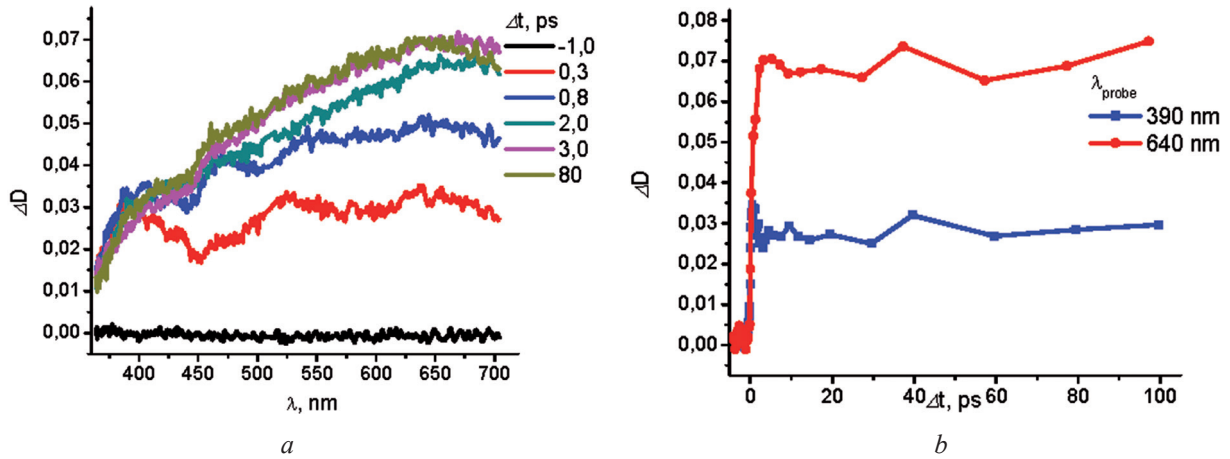

Fig. 4. Transient induced absorption spectra of LuAG:Ce single crystal at different time delays
between the pump and probe pulses (indicated) at pump pulse energy of 10 µJ (*a*);
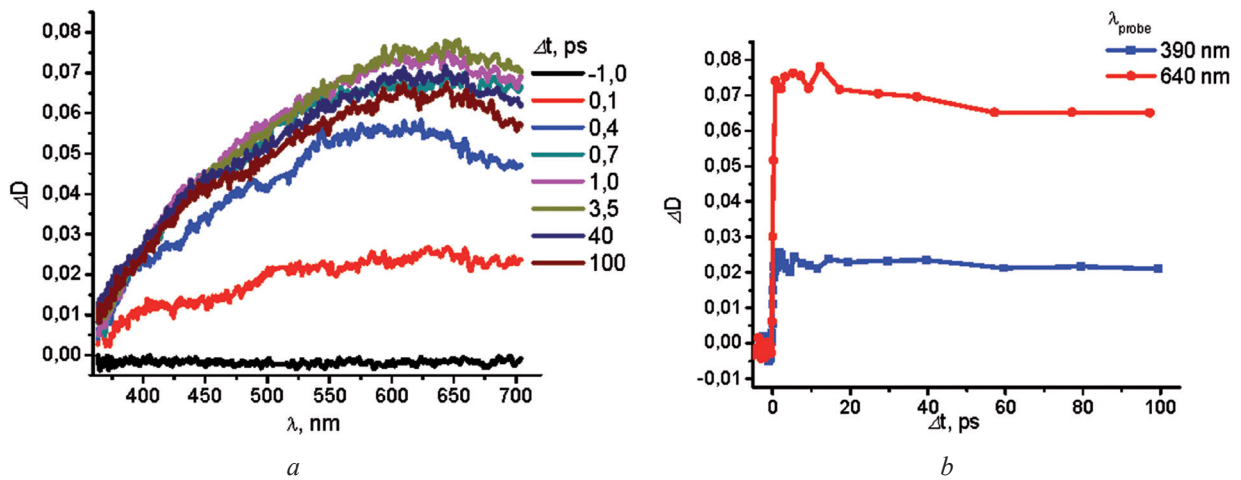and kinetics of probe beam absorption at the wavelengths of 390 and 640 nm (*b*)

Fig. 5. Transient induced absorption spectra of LuAG:Ce single crystal at different time delays
between the pump and probe pulses (indicated) at pump pulse energy of 10 µJ (*a*);
and kinetics of probe beam absorption at the wavelengths of 390 and 640 nm (*b*)
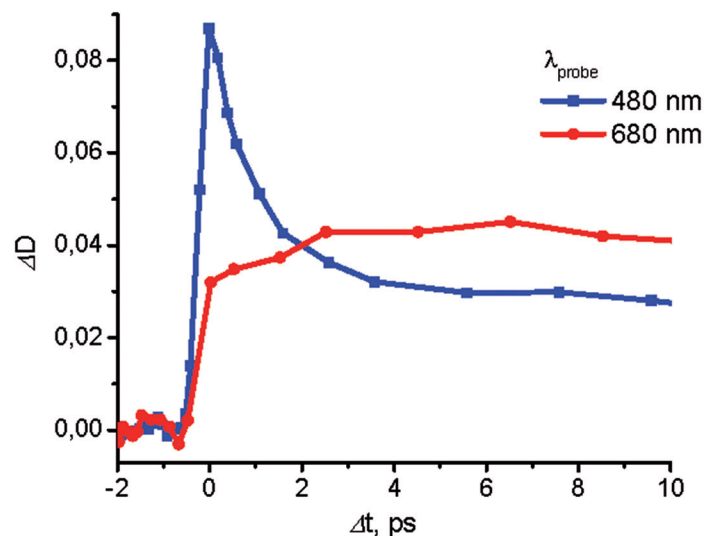
Fig. 6. Initial part of the kinetics of probe beam absorption at the wavelengths of 480 and 680 nm in GAGG:Ce

The spectra of GAGG:Ce contain two strongly overlapping wide bands: an absorption band peaked in the blue-green range (B1) and the second band peaking around 650 nm (B2). As seen in the differential absorption kinetics (parts (*b*) in Figs. 2–5), these bands appear simultaneously with the leading edge of the excitation. The B1 band (probed at 480 nm) shows an extremely fast decay component with the constant of about 2 ps and a slower decay with the constant of ~200 ps afterwards. The B2 band kinetics shows no fast component and decays with approximately the same time constant of ~200 ps as B1. The initial part of the differential optical density kinetics in GAGG:Ce is better shown within a reduced time range in Fig. 6.

The kinetics of transient absorption in the Mg-codoped GAGG:Ce is similar to that in the sample without codoping, except for the fast component B1 which becomes two times shorter and of smaller intensity than that in the corresponding Mg-free sample.

The B1 band is considerably less pronounced in the absorption spectra of LuAG:Ce and no fast decay component in the kinetics is observed for the differential optical density probed at the spectral wavelength of the B1 band.

Discussion. The existence of a fast decay component in the transient absorption of GAGG:Ce (see Figs. 2 and 3) was previously observed in [10]. The results presented in this work confirm its presence in GAGG and show that such component is absent in LuAG:Ce crystals, both uncodoped and with codoping with Mg^{2+} ions. This observation suggests that the transient absorption band peaking in the blue-green region cannot be associated with the excited radiating level of Ce^{3+}. Most probably, the B1 band is caused by free electron absorption, where the free electrons are generated via the absorption by Ce^{3+} and the subsequent transfer of the electrons to the conduction band. The narrow spectral width of the band might be attributed to the transitions of the free electrons to certain subband higher in the conduction band, which substantially enhances the resonant free electron absorption. The fast decay of the free electron absorption can be explained by the localization of free electrons at the traps in GAGG. Conversely, the B2 band is observed in all samples under study and can be explained as the absorption from the Ce^{3+} excited radiating levels. This interpretation is also supported by the different kinetic behavior of the B1 and B2 absorption bands observed in the GAGG:Ce sample at the initial stage after the short-pulse excitation (see Fig. 6). The equilibrium between the free electrons and the electrons at the excited Ce^{3+} level is established within approximately 2 ps after pump excitation, while the further decay of both free electron density (probed at 480 nm) and the population of the excited Ce^{3+} level (probed at 680 nm) proceeds at a similar rate, which is determined by radiative and nonradiative recombination processes. In the GAGG:Ce crystal codoped with magnesium, the relative contribution of the fast component is smaller and its decay time faster (about 1 ps) indicating that an additional recombination channel might have been introduced by codoping.

A difference in the rising time of the transient B2 absorption band kinetics between Mg-codoped and Mg-free garnets has also been observed as seen from Fig. 7.
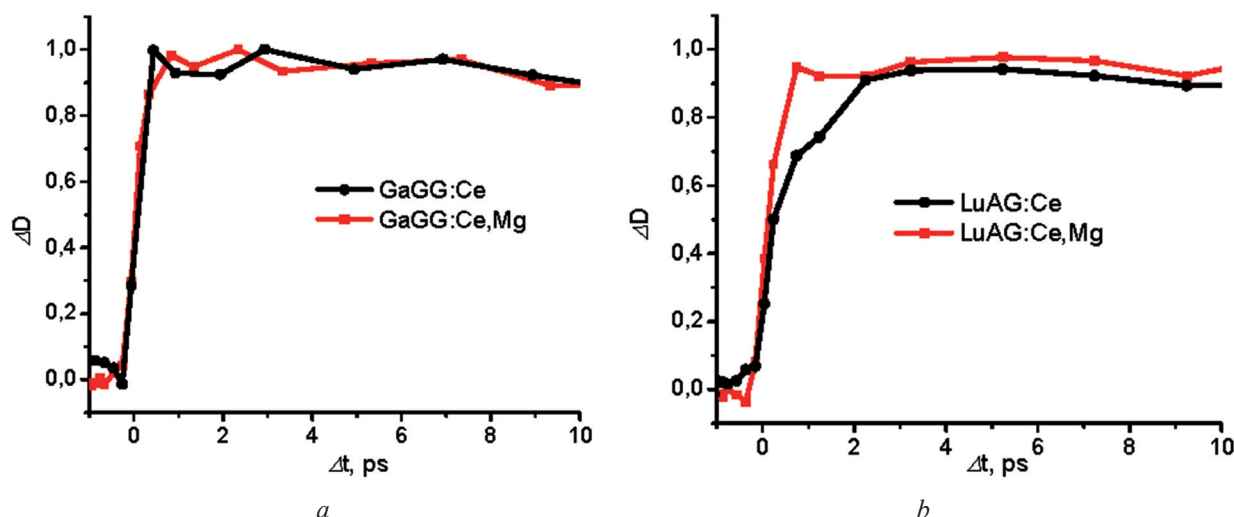
Fig. 7. Initial part of the kinetics of probe beam absorption at the wavelength 640 nm
for Mg-free and Mg-codoped GaGG:Ce (*a*) and LuAG:Ce (*b*) crystals

This difference is minimal in GAGG crystal but, in LuAG, the rising edge of the transient absorption response of the Mg-codoped crystals is considerably shorter due to capturing of free electrons from shallow traps. The Mg^{2+}-based defect centers, having a CT transition peaked at 265 nm, create deep trap levels approximately 1.5 eV below the bottom of conduction band. This can cause the same electrons to becaptured multiple times from shallow traps and prevents them from being re-captured by Ce^{3+} ions via the conduction band. Shallow traps capture electrons from the conduction band, while the thermal re-activation of the electrons back to the conduction band results in the population of Ce^{3+} radiating levels and is responsible for the delayed luminescence observed in such samples. In Mg-codoped crystals, this process is substantially suppressed by the re-capturing of trapped electrons by Mg-related centers and thus the re-population from the traps is less probable. As a result, the transient absorption decay is faster in the codoped crystals. Meanwhile, the additional channel of non-radiative recombination, which is introduced by Mg-codoping, reduces the light yield of garnet crystals.

Conclusions. Pump-probe method, using femtosecond laser sources, has been applied to study transient absorption of $Lu_3Al_5O_{12}$:Ce and $Gd_3Al_2Ga_3O_{12}$:Ce crystals with and without Mg-codoping. The 140fs pulses of pump (263 nm) were used to create free carriers in all the samples under study, whereas white super-continuum of the same duration was applied to measure transient absorption in the spectral range from 400 to 700 nm.

Difference in the dynamics of population of the excited state of the activator ions in two garnet-type scintillators: LuAG:Ce, and GAGG:Ce was observed. We assume that Mg-codoping introduces additional levels into the band gap, which facilitate transferring the trapped electrons back to the Ce ions. Two transient absorption bands were observed in doped and codoped crystals. The first band, present in all studied garnets, is broad and extends from approximately 450 nm over the whole visible region region and decays with characteristic time constant of approximately 200 ps. It is attributed to the absorption from the lowest excited state of Ce^{3+}. The second band is peaked at the blue-green region, emerges in subpicosecond domain and has the decay time of 1–2 ps. The time evolution of this band implies that it is caused by the light absorption by free electrons released to the conduction band. This band is most pronounced in GAGG:Ce, but is hardly detected in LuAG:Ce.

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