



Disorder response of $3d^3$ ions zero-phonon lines in the luminescence spectra of Yttrium-Aluminum-Gallium garnet solid solution ceramics



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ABSTRACT

The zero-phonon R-line luminescence spectra of $3d^3$ ions (Cr^{3+} and Mn^{4+}) in a series of $\text{Y}_3\text{Al}_{5-5y}\text{Ga}_y\text{O}_{12}$ ($0 \leq y \leq 1$) solid solution garnet ceramics were studied. Instead of increased inhomogeneous line broadening which is usually observed in solid solutions, discrete structure in the Cr^{3+} emission spectra is observed at lower values of y , whereas at high values of y the strong inhomogeneous broadening of the R-lines occurs and no discrete disorder response is observed. The results are explained based on the limited number of possible high-symmetry luminescent center geometries and the non-random preferential occupancy of tetrahedral lattice sites with gallium ions.

1. Introduction

The solid solution (mixed crystal) insulating crystals and ceramics doped with rare-earth and transition metal ions attract significant attention in optical spectroscopy studies. Solid solutions are especially interesting from the point of view of potential applications as scintillators, phosphors, laser materials and spectral hole-burning media because they allow variation of the physical properties of the material by variation of the composition. The structural disorder occurs in solid solutions due to the random distribution of ions in the crystal lattice sites. The inhomogeneous, i.e. related to the variations within the ensemble, broadening of electronic transitions (zero-phonon lines, ZPL) of impurity ions in solid solutions is, usually, significantly larger than that in the pure ordered crystals. This broadening was reported in literature [1,2] and is ascribed to the shift of the electronic energy levels in the random crystal field, connected to irregular structure of impurity ions surroundings. The inhomogeneous linewidths for impurity ions in solid solutions are usually similar by value to that observed in glasses of similar composition.

In our previous studies it was observed [3,4] that (in contrast to most studied materials) the modification of zero-phonon R-lines (${}^2\text{E}-{}^4\text{A}_2$) in the luminescence spectra of Cr^{3+} impurity ions in the concentration series of $\text{Lu}_{3x}\text{Y}_{3-3x}\text{Al}_5\text{O}_{12}$ ($0 \leq x \leq 1$) and $\text{Tb}_{3z}\text{Y}_{3-3z}\text{Al}_5\text{O}_{12}$ ($0 \leq z \leq 1$) garnet crystals occurs in a discrete rather than a continuous fashion and is not accompanied by strong inhomogeneous broadening. The effect was ascribed to high C_{3i} symmetry of octahedral $\text{Cr}_{\text{Al}}^{3+}$ sites

that allows only a limited number of non-equivalent Cr^{3+} centers in mixed environment of dodecahedral D_2 sites randomly occupied with ions of different size when the disorder is introduced into the Y/Lu/Tb sublattice. The observed dependences of energies and radiative lifetimes of ${}^2\text{E}$ states of locally identical (i.e. having identical configuration of nearest Y/Lu/Tb cations) Cr^{3+} centers inside different LuYAG/TbYAG hosts on Lu content x and Tb content z were explained by lattice compression and dilation occurring according to Vegard's law.

The purpose of the study presented here is to trace the modification of the zero-phonon R-line spectrum of garnet solid solutions in a more complicated situation of the disorder introduced into the sublattices of tetrahedral and octahedral Al/Ga sites. We report the results of the luminescence spectroscopy studies of $3d^3$ ions (Cr^{3+} and Mn^{4+}) zero-phonon lines in $\text{Y}_3\text{Al}_{5-5y}\text{Ga}_y\text{O}_{12}$ solid solution garnet ceramics.

2. Experiment

The experiments were performed with a series of solid solution $\text{Y}_3\text{Al}_{5-5y}\text{Ga}_y\text{O}_{12}$ garnet ceramics ($0 \leq y \leq 1$). The garnet ceramics samples were prepared at Philips Research Eindhoven by sintering a mixture of base oxides of 4N-purity in air atmosphere in the form of pills of 14 mm diameter and 1–2 mm thickness. Based on the X-ray diffraction patterns it was concluded that all samples consist of a single garnet phase.

Cr^{3+} luminescence spectra were recorded taking advantage of the trace amounts of Cr present in the samples that were not intentionally

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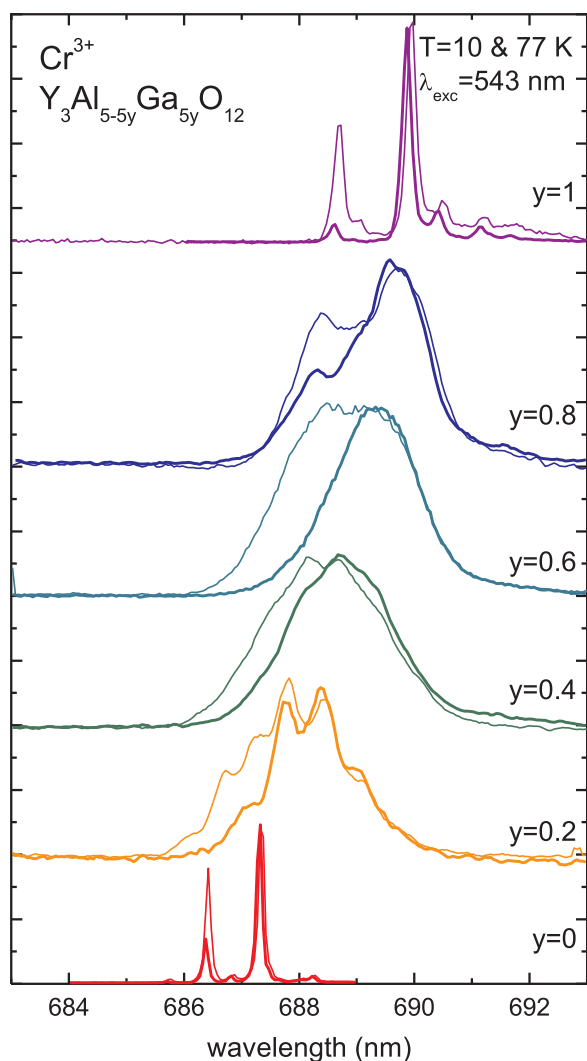


Fig. 1. Fluorescence spectra of Cr^{3+} ions in $\text{Y}_3\text{Al}_{5-5y}\text{Ga}_y\text{O}_{12}$ ceramics samples. $\lambda_{\text{exc}} = 543$ nm, thick lines: $T = 10$ K, thin lines: $T = 77$ K.

doped with chromium. These samples were doped with 0.2 at% of cerium (they were originally synthesized for scintillator studies). Mn^{4+} luminescence spectra were obtained with the samples doped with 0.1 mol% Mn without charge compensation; in these samples the majority of Mn dopant ions were in the 3+ valence state.

For optical measurements the samples were placed on the cold finger ($T = 10$ K) of a liquid helium refrigerator or immersed in liquid nitrogen ($T = 77$ K). The Cr^{3+} and Mn^{4+} luminescence was excited with diode-pumped solid state (DPSS) lasers operating at 543 or 473 nm respectively. The luminescence was detected with a double-grating monochromator (0.01 nm resolution) and a photomultiplier operating in a photon-counting mode.

3. Experimental results

The luminescence R-line (${}^2\text{E}-{}^4\text{A}_2$) spectra of Cr^{3+} ions for the series of $\text{Y}_3\text{Al}_{5-5y}\text{Ga}_y\text{O}_{12}$ solid solution garnet ceramics excited at $\lambda_{\text{exc}} = 543$ nm via the ${}^4\text{T}_2$ absorption band are shown in Fig. 1 ($T = 10$ and 77 K). At low temperatures the $\lambda_{\text{exc}} = 543$ nm light does not excite Ce^{3+} ions even taking into account the effect of Ce^{3+} excitation at energies below the zero-phonon line [5], so only the Cr^{3+} ions luminescence is observed.

The spectra of the ceramics samples with $y = 0$ and $y = 1$ correspond to the spectra of YAG:Cr [6,7] and YGG:Cr [6,8,9] respectively.

The narrow R-line (${}^2\text{E}-{}^4\text{A}_2$) emission is observed around 690 nm and is split in a doublet (R_1 and R_2 line) due to a splitting of the ${}^2\text{E}$ excited state. The spectra observed are similar to those in single-component garnet crystals. The additional weak lines observed close to the R-lines in YAG:Cr and YGG:Cr are due to the phenomenon of inversion between Y^{3+} and $\text{Al}^{3+}/\text{Ga}^{3+}$ cations in dodecahedral and octahedral sites of the garnet lattice [10]. This inversion produces local distortions of the crystalline lattice that could perturb the ideal structure of the neighbouring cationic sites.

The behavior of the Cr^{3+} spectra in $\text{Y}_3\text{Al}_{5-5y}\text{Ga}_y\text{O}_{12}$ samples with the change of y at its lower values is similar to that reported in [3] for $\text{Lu}_{3x}\text{Y}_{3-3x}\text{Al}_5\text{O}_{12}$: a discrete structure is observed when the smaller aluminum ions are one by one replaced with larger gallium ones. At $T = 10$ K the luminescence lines correspond mostly to the lower-energy (R_1) components of the doublet ${}^2\text{E}-{}^4\text{A}_2$ transitions, though some contribution of partially occupied higher-energy (R_2) transition is still present, at $T = 77$ K the contribution of R_2 transitions becomes more significant due to increased thermal (Boltzmann) population.

The similarity between the Cr^{3+} R-line spectra of $\text{Y}_3\text{Al}_{5-5y}\text{Ga}_y\text{O}_{12}$ at lower values of y ($y = 0.2$) and the discrete spectrum of $\text{Lu}_{3x}\text{Y}_{3-3x}\text{Al}_5\text{O}_{12}$ ($x = 0.3$) is shown in Fig. 2; note the different (top and bottom) wavelength scales for the two spectra. A good correspondence of the two spectra is achieved when the wavelength scales are not only shifted, but the scale for $\text{Lu}_{3x}\text{Y}_{3-3x}\text{Al}_5\text{O}_{12}$ is expanded by a factor of 1.43. This is a manifestation of larger relative difference between the ionic radii of tetrahedral Ga and Al (0.47 Å and 0.39 Å) and between dodecahedral Y and Lu (1.019 Å and 0.977 Å) whereas the distances between the Cr^{3+} ion on a octahedral site and the tetrahedral Ga/Al and dodecahedral Y/Lu ions are the same. As a result, replacement of a nearby tetrahedral Al by Ga causes a larger shift in R-line energy than the replacement of nearby dodecahedral Y by Lu (12.3 cm^{-1} vs. 8.6 cm^{-1}).

At high values of y the behavior of the Cr^{3+} spectra in $\text{Y}_3\text{Al}_{5-5y}\text{Ga}_y\text{O}_{12}$ samples with the change of y is different from the discrete spectra observed at ($y = 0.2$) and from the discrete spectra of the $\text{Lu}_{3x}\text{Y}_{3-3x}\text{Al}_5\text{O}_{12}$ series [3]. The strong inhomogeneous broadening of the R-lines occurs and no discrete disorder response is observed.

Mn^{4+} ions have the same $3d^5$ electronic configuration as Cr^{3+} , so it

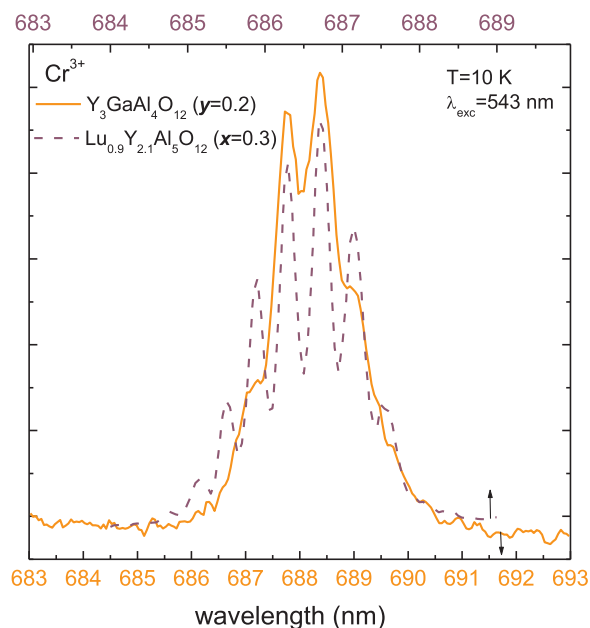


Fig. 2. Comparison of the R-line fluorescence spectra of Cr^{3+} ions in $\text{Y}_3\text{Al}_{5-y}\text{Ga}_y\text{O}_{12}$ ($y = 0.2$) ceramics and $\text{Lu}_{3x}\text{Y}_{3-3x}\text{Al}_5\text{O}_{12}$ ($x = 0.3$) crystal [3]. $T = 10$ K. Top and bottom wavelength axes are different for the two spectra (correspondence indicated by arrows).

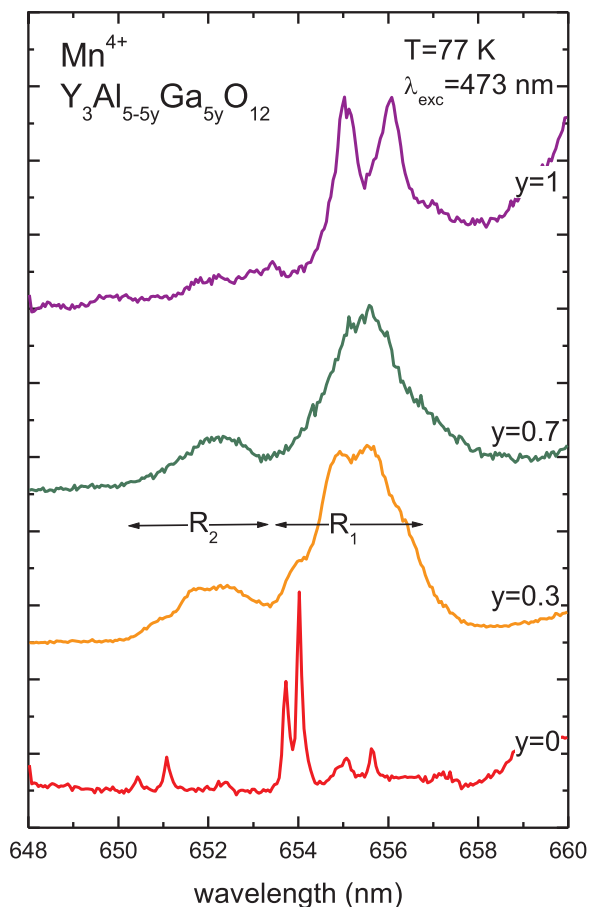


Fig. 3. Fluorescence spectra of Mn^{4+} ions in $\text{Y}_3\text{Al}_{5-y}\text{Ga}_y\text{O}_{12}$ ceramics samples. $\lambda_{\text{exc}} = 473$ nm, $T = 77$ K.

is interesting to compare the R-line (${}^2\text{E} \rightarrow {}^4\text{A}_2$) spectra of Mn^{4+} and Cr^{3+} ions in the series of $\text{Y}_3\text{Al}_{5-y}\text{Ga}_y\text{O}_{12}$ solid solution garnet ceramics. Though in the Mn-doped ceramics samples the luminescence spectra were dominated by the broadband Mn^{3+} luminescence [11], under the $\lambda_{\text{exc}} = 473$ nm excitation it was still possible to detect Mn^{4+} R-line spectra which are shown in Fig. 3. The spectrum of the ceramics sample with $y = 0$ corresponds to the spectrum of $\text{YAG}:\text{Mn}^{4+}$ [12,13] with much larger (60 cm^{-1}) R_1 - R_2 splitting than that for Cr^{3+} (19 cm^{-1}), so the sets of R_1 and of R_2 lines do not overlap in the spectra. The behavior of the Mn^{4+} spectra with changing y is somewhat similar to that of Cr^{3+} ones: some structure is visible at $y = 0.3$ whereas at $y = 0.7$ the strong inhomogeneous broadening of the R-lines obscures any discrete structure. The additional complication of the spectra is introduced by the significant contribution of Mn^{4+} multisites [13] that are probably due to local charge compensation of Mn^{4+} . Nevertheless the conclusion about different behavior of the spectra with changing y at low and high y may be done.

4. Discussion

In [3] the discrete modification of R-line spectra of Cr^{3+} in LuYAG crystals with the change of Lu/Y ratio was explained by the high symmetry of Cr^{3+} center in the crystal lattice. In the cubic O_h garnet lattice Cr^{3+} ions replace the Al^{3+} ions in the octahedral sites of a C_{3i} symmetry and are surrounded by six oxygen ions [6,7]. In the next cation coordination sphere the six Lu/Y ions nearest to the Cr^{3+} site also form an octahedron with C_{3i} symmetry with a Cr^{3+} ion in its center [3]. It follows from the C_{3i} symmetry (including trigonal axis and inversion) that all six Lu/Y positions are equivalent in relation to Cr^{3+} . For the octahedrally-coordinated Cr^{3+} site in $\text{Y}_3\text{Al}_{5-y}\text{Ga}_y\text{O}_{12}$ solid

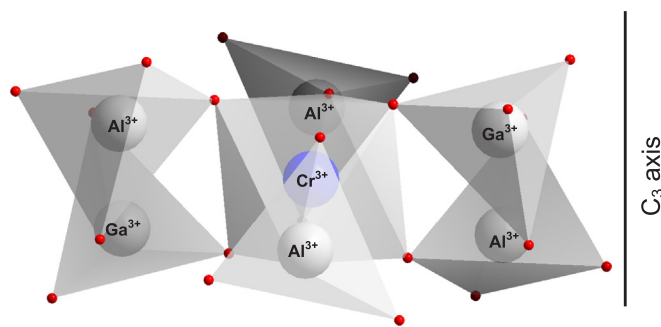


Fig. 4. Al/Ga tetrahedra environment of an octahedral $\text{Cr}_{\text{Al}}^{3+}$ center with C_{3i} symmetry in the garnet lattice. One of three possible Cr^{3+} ($2\text{Ga}4\text{Al}$) centers is shown. C_3 is the trigonal symmetry axis. For clarity oxygen ions are shown as small spheres and dodecahedral Y sites are omitted.

solution the nearest randomly occupied Al/Ga sites are tetrahedral and form an octahedron with C_{3i} symmetry around this site – see Fig. 4. Thus in full analogy with the consideration for LuYAG [3] it can be concluded that in $\text{Y}_3\text{Al}_{5-y}\text{Ga}_y\text{O}_{12}$ there is a limited number of different surroundings of a $\text{Cr}_{\text{Al}}^{3+}$ center by tetrahedrally-coordinated Al/Ga ions. We will label the Cr^{3+} centers surrounded with m gallium and n aluminum ions as $\text{Cr}^{3+}(m\text{Ga}n\text{Al})$ centers ($m+n = 6$). In pure YAG all six nearest tetrahedrally-coordinated cations are aluminum ions ($\text{Cr}^{3+}(6\text{Al})$ center); the replacement of Al^{3+} ions one by one with Ga^{3+} in solid solutions produces a few types of Cr^{3+} centers: one $\text{Cr}^{3+}(1\text{Ga}5\text{Al})$, three non-equivalent $\text{Cr}^{3+}(2\text{Ga}4\text{Al})$ centers, three $\text{Cr}^{3+}(3\text{Ga}3\text{Al})$, three $\text{Cr}^{3+}(4\text{Ga}2\text{Al})$, one $\text{Cr}^{3+}(5\text{Ga}1\text{Al})$, and one $\text{Cr}^{3+}(6\text{Ga})$. The total number of possible non-equivalent Cr^{3+} centers is 13. Each additional larger Ga ions introduced into the Cr^{3+} surrounding shifts the R line towards longer wavelengths. The statistics of occurrence of different $\text{Cr}^{3+}(m\text{Ga}n\text{Al})$ centers determines the shape of the R-line spectrum at different Ga content y . The octahedrally-coordinated Al/Ga ions are about 1.5 times further from the $\text{Cr}_{\text{Al}}^{3+}$ center than the tetrahedrally-coordinated ones, so the influence of the disorder in the octahedrons sublattice on the Cr^{3+} R-line spectra is expected to be significantly less.

The above considerations explain the discrete behavior of the Cr^{3+} R-line spectra of $\text{Y}_3\text{Al}_{5-y}\text{Ga}_y\text{O}_{12}$ at lower values of y as well as the similarity of the spectrum to that of LuYAG crystal clearly visible in Fig. 2. Indeed, the replacement of smaller Al ions with larger Ga ions in the six tetrahedral cation sites around Cr^{3+} yields the qualitatively same effect as the replacement of smaller Lu ions with larger Y ions in the six surrounding dodecahedral cation sites.

The different behavior of the spectra at higher values of y (Fig. 1) requires different explanation. Indeed, in $\text{Lu}_{3x}\text{Y}_{3-3x}\text{Al}_5\text{O}_{12}$ crystals both the replacement of a fraction of (smaller) lutetium ions with (larger) yttrium ones in LuAG and vice versa in YAG result in the clear discrete series of R-lines (Fig. 2 in [3]). Similar behavior should be expected for $\text{Y}_3\text{Al}_{5-y}\text{Ga}_y\text{O}_{12}$ while replacing a fraction of (smaller) aluminum ions with (larger) gallium ones in YAG and vice versa in YGG assuming random distribution of Al and Ga ions in the tetrahedral sites sublattice. Contrary to such expectation, replacement of a fraction of gallium ions in YGG with Al ions results in inhomogeneous broadening of the R-lines without any discrete disorder response – Fig. 1. Also, the overall extent of the broadening (spectral width) is smaller, which can be observed by comparing the spectra for $y = 0.2$ and $y = 0.8$ in Fig. 1. Such R-lines behavior suggests non-random distribution of Al and Ga ions in the tetrahedral sites sublattice. The observations can be explained when it is assumed that at high values of y Al ions do not occupy tetrahedral sites statistically, but preferentially replace Ga ions in octahedral sites. This preferential replacement of gallium ions in YGG with Al ions in octahedral sites, retains the purely “tetrahedral Ga” surroundings of the $\text{Cr}_{\text{Al}}^{3+}$ luminescent probe ions up to significant values of Al content ($1-y$). As a result, the R-lines of $\text{Cr}^{3+}(6\text{Ga})$ centers are observed in a wide

range of the values of y . The observed inhomogeneous broadening of the Cr^{3+} (6Ga) R- lines of may be ascribed to the disorder in the octahedral Al/Ga sublattice: the octahedral Al/Ga ions are located about 1.5 times further from a given octahedral Cr^{3+} (6Ga) center than the tetrahedral ones and provide more possible configurations. As a result of weaker influence of ions substitution in octahedral positions and of more numerous different R-line positions due to disorder in these positions the structureless broadening occurs and the overall spectral broadening is less.

The preferential occupation of tetrahedral sites by Ga and of octahedral sites by Al also clearly follows from the spectra presented in Fig. 2. The spectrum of $\text{Y}_3\text{Al}_{5-5y}\text{Ga}_{5y}\text{O}_{12}$ at low value of larger-size Ga ions content $y = 0.2$ is similar to the spectrum of $\text{Lu}_{3x}\text{Y}_{3-3x}\text{Al}_5\text{O}_{12}$ with larger-size Y ions content $(1-x) = 0.7$. As soon as the distribution of Lu/Y ions in LuYAG is supposed to be random, the preferential (non-random) occupation of tetrahedral sites in $\text{Y}_3\text{Al}_{5-5y}\text{Ga}_{5y}\text{O}_{12}$ with Ga ions is necessary to explain the discrepancy in larger ions content which accounts for the spectra compared in Fig. 2.

The surprising conclusion about the preferential occupation of the smaller tetrahedral sites by larger Ga ions and of larger octahedral sites by smaller Al ions is in full agreement with the results of x-ray diffraction [14,15] and NMR [16] studies, where the same conclusion was made. The effect is explained [15] by the greater covalency of the Ga-O bonds than that of the Al-O bonds. It should be noted that the preferential occupancy of tetrahedral sites with Ga ions may be different for the tetrahedra surrounding the octahedral Al^{3+} site and the octahedral $\text{Cr}_{\text{Al}}^{3+}$ center. The stronger preference in occupation of tetrahedral sites with Ga ions around the octahedral $\text{Cr}_{\text{Al}}^{3+}$ center may further contribute to the “asymmetry” of the R-line spectra response to the variations of Ga content at high and low values of y .

In Fig. 1 it may be seen that the shift of the inhomogeneously-broadened R-lines of Cr^{3+} (6Ga) centers with the decrease of y at high Ga content occurs in the shorter-wavelength direction. In contrast, the shift of the R-lines of locally-identical Cr^{3+} centers observed in [3,4] when the larger ions were replaced by smaller ones occurred in longer-wavelength direction (opposite to the shift of the whole R-line set barycenter) and was explained by the whole lattice compression according to Vegard's law [3,4,17]. This effect may be ascribed to the peculiarities of lattice distortions at intermediate distances in conditions when significant deviations from Vegard's law occur [15].

The results obtained for the Mn^{4+} ions (Fig. 3) are qualitatively similar to that for Cr^{3+} ones and thus further confirm the suggested interpretation.

5. Conclusions

In $\text{Y}_3\text{Al}_{5-5y}\text{Ga}_{5y}\text{O}_{12}$ garnet solid solutions disorder is introduced into both sublattices of tetrahedral and octahedral Al/Ga sites. It was observed experimentally that the modification of zero-phonon R-lines spectra of Cr^{3+} impurity ions in the concentration series of

$\text{Y}_3\text{Al}_{5-5y}\text{Ga}_{5y}\text{O}_{12}$ ($0 \leq y \leq 1$) solid solution ceramics occurs differently at high Al and high Ga content. Discrete Cr^{3+} R-line spectra are observed at lower values of y whereas at high values of y inhomogeneous broadening of the R-lines occurs and no discrete disorder response is visible. The effect may be explained by non-random distribution of Al and Ga ions in the tetrahedral sites sublattice, namely, the preferential occupation of tetrahedral sites with Ga ions. It may be concluded that luminescence spectroscopy of probe Cr^{3+} ions in solid solutions enables to detect the variations in the occupancy of sublattices by different ions and to reveal their non-random distribution. The R-line spectra of Mn^{4+} impurity ions in $\text{Y}_3\text{Al}_{5-5y}\text{Ga}_{5y}\text{O}_{12}$ show the same difference in behavior with changing y at its low and high values and thus confirm the conclusion about non-random distribution of Al and Ga ions between the tetrahedral and octahedral sites sublattices.

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