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Photoluminescence and structural characterization of $MgAl_2O_4$ irradiated with swift Bi ions

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ABSTRACT

The optically stimulated luminescence in $MgAl_2O_4$ single crystals associated with defects produced by high energy (670 MeV) Bi ions with electronic stopping power $S_e = 36.6$ keV/nm has been studied. Such energy loss is almost five times higher than the threshold for latent track formation in spinel, about 7.5 keV/nm. High resolution transmission electron microscopy (HRTEM) examination has revealed that ion tracks in the sub-surface layer of irradiated specimens retain a crystalline structure. Photoluminescence (PL) spectra from the near surface region were recorded during excitation at 355 nm, 473 nm and 532 nm using a laser confocal microscopy technique. It was found that PL spectra were always composed of broad overlapping bands positioned over the entire spectral range from the excitation wavelengths till 850 nm. It is suggested that such spectral composition might be due to a high concentration of antisite defects with large variation of the Mg/Al ratio in the disordered regions surrounding ion track cores. PL measurements on 23 MeV Ne ($S_e = 3.5$ keV/nm) ion irradiated spinel, when no latent tracks are formed, have produced similar spectra as those for Bi ions. This indicates the same nature of defect-related luminescent centers formed at significantly different levels of electronic stopping power – below and above the threshold for latent track formation. The conclusion and findings may provide new insights for interpreting the optical spectra in this material.

1. Introduction

Radiation-induced changes in the microstructure and optical properties of $MgAl_2O_4$ remain the subject of continuous interest over the last few decades. Compared to alumina, it has excellent resistance to neutron irradiation, a property that is not common in oxide ceramics [1–3]. Demonstrating exceptional stability during accumulation of defects produced in elastic collisions, spinel is rather sensitive to damage via relaxation of dense electronic excitations and has a relatively low threshold for latent track formation, about 7.5 keV/nm [4,5]. The morphology of ion track regions in $MgAl_2O_4$ irradiated with swift heavy (350 MeV) Au and (200 MeV) Xe ions was studied in detail using transmission electron microscopy techniques (TEM) and high angular resolution electron channeling X-ray spectroscopy (HARECXS) [6–9]. Yasuda and et al. [8] reported a phase transformation to a defective rock-salt structure inside the track core. Cation disordering (Al and Mg ions exchanged their positions) and strained regions were detected around the defective structure. It should be noted that no amorphization was observed inside the ion track core for electronic stopping power 35 keV/nm deposited by 350 MeV Au ions [6].

The optical properties of spinel irradiated with swift heavy ions (SHI) have only very recently begun to be studied [10–14]. Detailed analysis of defect accumulation in $MgAl_2O_4$ irradiated with 152 MeV Xe and 2.24 GeV Au ions was undertaken in Ref. [10] using absorption spectroscopy and EPR. Defect concentration increased with fluence up to 2×10^{12} cm^{-2} with no detectable saturation. The authors have obtained

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thermal annealing curves for five (4.8, 5.3, 5.9, 6.6 and 7.0 eV) absorption bands and concluded that the 5.9 and 6.6 eV bands were related to complexes of defects, which include antisite defects and Al^{3+}, Mg^{2+} vacancies; the 7.0-eV band was assigned to the formation of exciton-like states [11]. It is well-known that the 4.8 and 5.3 eV bands are associated with single electronic F^+- and F-centers, respectively [15,16]. At the same time, there is no mention of aggregate color center formation. Optical absorption, photoluminescence and confocal Raman spectroscopy have been used to characterize defects induced in spinel by 220 MeV Xe ions [13,14]. Vacuum ultraviolet spectroscopy indicated the formation of intrinsic optically active centers with emission band peaked at 1.7 eV. It was demonstrated that there is an energy transfer from the excited F^+ center to the new center. Additionally, an excitation spectrum registered a band at 3.4 eV, which directly refers to the excited state of the new center.

The data on confocal PL and time-correlated single photon counting studies of 152 MeV Xe ion irradiated $MgAl_2O_4$ single crystals were presented in Ref. [17]. It was shown that radiation defects produced by swift xenon ions give rise to a broad luminescence band positioned in the 500–800 nm wavelength range. Measurements of the PL decay curves have shown that the lifetime of the defect-related PL signal gradually increases from 6 to 13 ns in the 500–650 nm spectral range and decreases with increasing ion fluence. The decrease of the luminescence intensity observed after fluences of $\sim 10^{13}$ cm^{-2} was assigned to the start of overlapping of individual ion track core regions.

In the present work we report on the PL and TEM examination of $MgAl_2O_4$ single crystals, irradiated with 670 MeV Bi ions with surface electronic stopping power 36.6 keV/nm, the highest level of energy deposition used investigated to date. The aim of these experiments is at further elucidation the mechanisms of defects formation processes in $MgAl_2O_4$ induced by swift heavy ions impact.

2. Experiment

In this work we have used (100) oriented single crystalline stoichiometric magnesium aluminate spinel $MgAl_2O_4$ with thickness of 500 μm purchased from CRYSTAL GmbH. The specimens were irradiated with 670 MeV Bi ions in the range of fluences from 10^{10} to 10^{12} cm^{-2} at room temperature at the U-400 FLNR JINR cyclotron, Dubna. The average Bi ion flux was 5×10^7 cm^{-2} s^{-1}. Ion beam homogeneity over the irradiated specimen surface was controlled using beam scanning in the horizontal and vertical directions and was better than 10 %. A portion of spinel specimens was also irradiated with 100 MeV Kr ions to fluences from 10^{12} cm^{-2} to 1.9×10^{13} cm^{-2} and 23 MeV Ne ions (10^{13} cm^{-2}, 2×10^{13} cm^{-2}, 6×10^{13} cm^{-2}) at the IC-100 cyclotron with average ion flux 5×10^8 cm^{-2} s^{-1}. Such Bi, Kr and Ne ion fluxes did not induce any significant (no more than 10°) heating of irradiated crystals.

TEM lamellae were prepared by means of focused ion beam (FIB) lift-out using an FEI Helios Nanolab 650. To minimize possible ion beam induced damage (by FIB), the samples were polished with Ga ion energies down to 1 keV. Structural examination was done with a JEOL ARM200F operated at 200 kV at the Centre for High Resolution Transmission Electron Microscopy, Nelson Mandela University (Port Elizabeth, South Africa).

Luminescence spectra were registered using a Nanofinder High End (LOTIS TII Japan-Belarus) spectrometer combined with a 3D scanning confocal microscope. Excitation was provided by UV (355 nm), blue (473 nm) and green (532 nm) lasers with powers of 2 mW and a spot size about of 1 μm. PL spectra were gathered using a backscattering set-up from depth no more than 3 μm, i.e. in the target layer with the dominant contribution of electronic stopping power in energy losses of both Bi and Kr ions as one can see from Fig. 1, where energy loss profiles calculated using SRIM software [18] for density 3.58 g/cm^3 are given together with schematic imaging of the PL measurements.

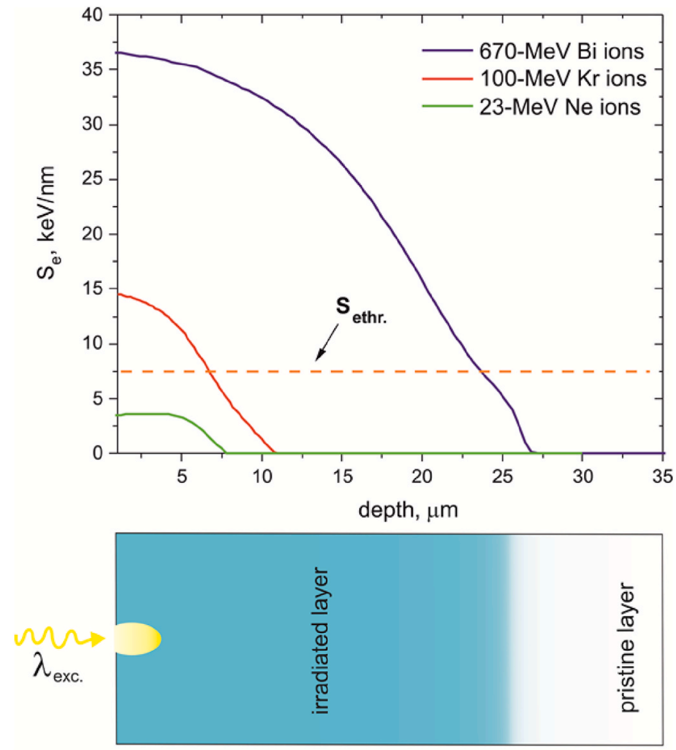

Fig. 1. Ionization energy loss profiles of 670 MeV Bi, 100 MeV Kr and 23 MeV Ne ions in $MgAl_2O_4$ and scheme of the PL experiments.

3. Results and discussion

TEM examination in both planar and cross-section geometry has demonstrated that Bi ions produce latent tracks in the near surface layer of spinel crystals affected by 36.6 keV/nm energy deposition in the electron subsystem. A TEM image of Bi ion induced tracks in $MgAl_2O_4$ is shown in Fig. 2ab. Fig. 2a shows a high resolution (HR) TEM micrograph of a single 670 MeV Bi ion track in $MgAl_2O_4$. Ion track remains crystalline, but the structure is perturbed leading to a visible strain field. Fig. 2b shows a bright field (BF) TEM micrograph of a larger region irradiated to a fluence of 10^{11} ions/cm^2, the tracks are strongly diffracting in this orientation confirming their crystalline nature despite several visible partially overlapped tracks.

The difference in the structure of spinel irradiated with Ne and Bi ions at ionizing energy losses below and above the threshold of latent track formation is demonstrated in cross-sectional TEM images (Fig. 2cd). Below the threshold, the damage structure is dominated by randomly distributed defect clusters with no clear evidence of the ion trajectory. This structure suggests that TEM visible defect clusters are formed primarily in locations where the local energy deposition is maximized through a combination of nuclear collision cascades and the local electronic energy deposition. This is in stark contrast to the structure observed in (d). The ion trajectory is clearly visible as the defects align along the ion path creating a near continuous extended structure. Such a structure is consistent with the process of dense electronic excitation where inelastic energy deposition is relatively constant over several tens of nanometers and nuclear collision cascades are improbable.

Fig.3abc show luminescence spectra taken from spinel specimens irradiated with Bi ions to several fluences during pumping at 355 nm, 473 nm and 532 nm. The PL spectra measured from Ne and Kr ions irradiated crystals are given in Fig. 3(d and e,f). The spectra were recorded after photobleaching by excitation laser for 250 s. During this time, the integral PL intensity decreased about 2.7 times and reached a constant level.

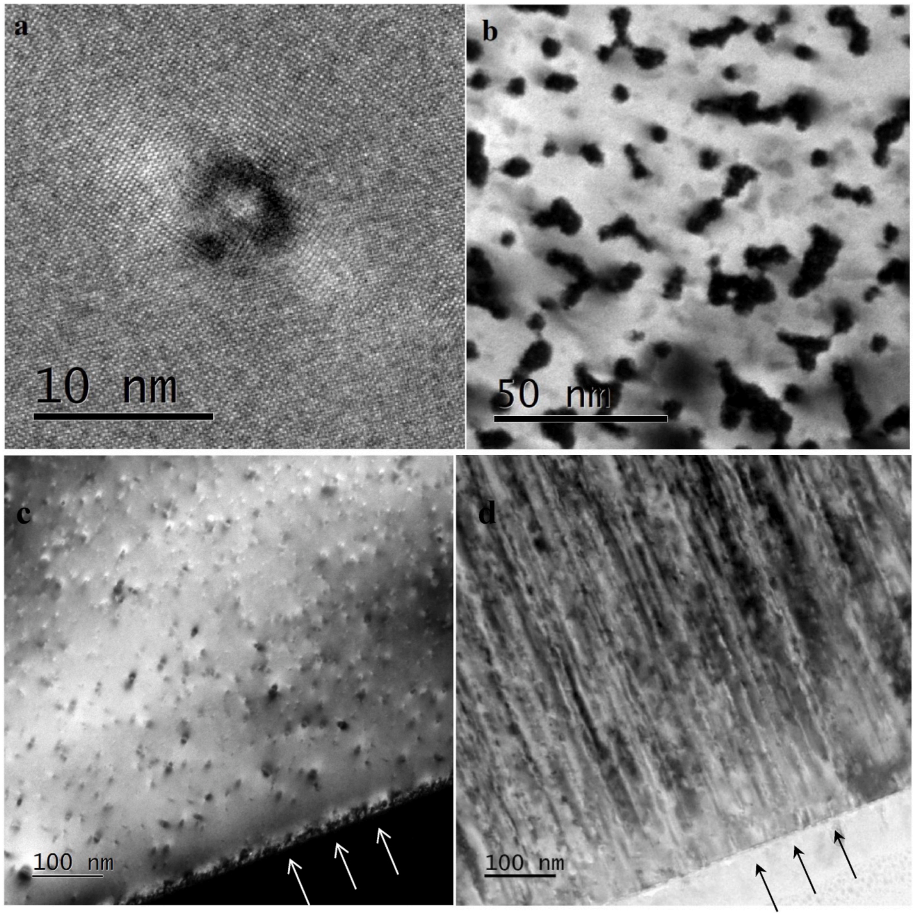

Fig. 2. Plan view HRTEM (a) and BF TEM (b) image of 670 MeV Bi ion induced tracks in $MgAl_2O_4$. Cross-sectional BF TEM images of defect structure in $MgAl_2O_4$ irradiated with 6×10^{12} cm^{-2} 23 MeV Ne (c) and 10^{12} cm^{-2} 670 MeV Bi (d) ions. The arrows show the direction of beam incidence.

The excitation with UV and blue light of intact material leads to an emission band with a maximum of 520 nm and a series of lines in the region of 680–720 nm. According to the literature data, the broad band peaked at 520 nm and excited by all lasers except 532 nm is due to Mn^{2+} impurity ions [19,20] and the lines in the red region of the spectrum are associated with trivalent chromium impurities [21,22]. Such spectral composition for pristine material is typical of luminescence excited both by electromagnetic radiation and by charged particles - electrons and low energy heavy ions [23,24].

Clearly, all PL spectra regardless of excitation are characterized by one common distinguishing feature – they are continuous and registered over the entire spectral range from the excitation wavelength till detection limit, about 800 nm. A very similar continuous structureless emission band generated by 2.78 eV (445 nm) photons has been observed recently in 152 MeV Xe ion irradiated $MgAl_2O_4$ [17]. Overlapped broad bands with maxima at 218, 245, 318, 370 and 428 nm were detected during irradiation with high energy (609, 425, 245 and 93 MeV) Xe^{q+} ions [25]. The first three bands are ascribed to localized excitons, F and F^+-centers, while the rest relate to excitons bound with cations (370 nm), and oxygen ions (428 nm) [25]. At the same time, contrary to PL, the ion beam induced luminescence spectra do not have bands/lines in red and near the infrared range except for those related to Mn^{2+} and Cr^{3+} impurities. These bands were also not detected when excited by low-energy ions (320 keV Ar) as well as during cathodoluminescence measurements [23,24]. This suggests a low efficiency of energy transfer to the corresponding luminescence centers by charge carriers compared to direct in-center excitation. A similar situation was observed for the 540 nm luminescence band in Al_2O_3 attributed to F_2^{2+}-centers. It was practically undetectable under irradiation with high-energy heavy ions [26]. Broad emission band in the spectral range of interest for us was registered in PL spectra after neutron [27–30] and electron irradiation and after pulsed plasma-ion modification [31]. The nature of these band associated with radiation defects was not established.

A broad and long tailed luminescence band with maximum at 420 nm has been obtained from powdered pristine spinel samples during excitation at 365 nm [32]. Its origin was associated with intrinsic lattice defects, namely with antisite defects formed when Mg substitutes Al and vice versa. This results in positively and negatively charged lattice sites in spinel due to its ability to tolerate cation disorder in tetrahedral and octahedral positions. One can note that the PL spectrum shown in Fig. 1 in Ref. [32] also has an indication of the contribution of a band with a maximum at 650 nm in the total luminescence signal. As demonstrated in Ref. [33], the emission in the region around 650 nm, which is definitely one of the dominant features for specimens irradiated with Bi ions (Fig. 3), is typical for $MgAl_2O_4:Mn^{4+}$ phosphor synthesized by the co-precipitation method and its parameters correlate with the anti-site Mg ⇔ Al disorder degree. The relation of the emission at 650 nm to processes involving Mn^{4+} and Mn^{3+} ions has been shown also by Kazarinov et al. who identified six luminescence channels related to the Mn ions incorporated in the $MgAl_2O_4$ spinel structure [34] and producing light in bands peaked at 520, 651, 733, and 926 nm.

Combined experimental and theoretical results of analysis of the PL spectra from spinel with different Mg/Al composition obtained after annealing at different temperatures through a sol-gel combustion route, were presented in Ref. [35]. As was found, the emission spectra during excitation at 250 nm may be composed of ten bands, which can be linked to different types of defect centers, such as F, F_2, F^+, F_2^{2+} and different

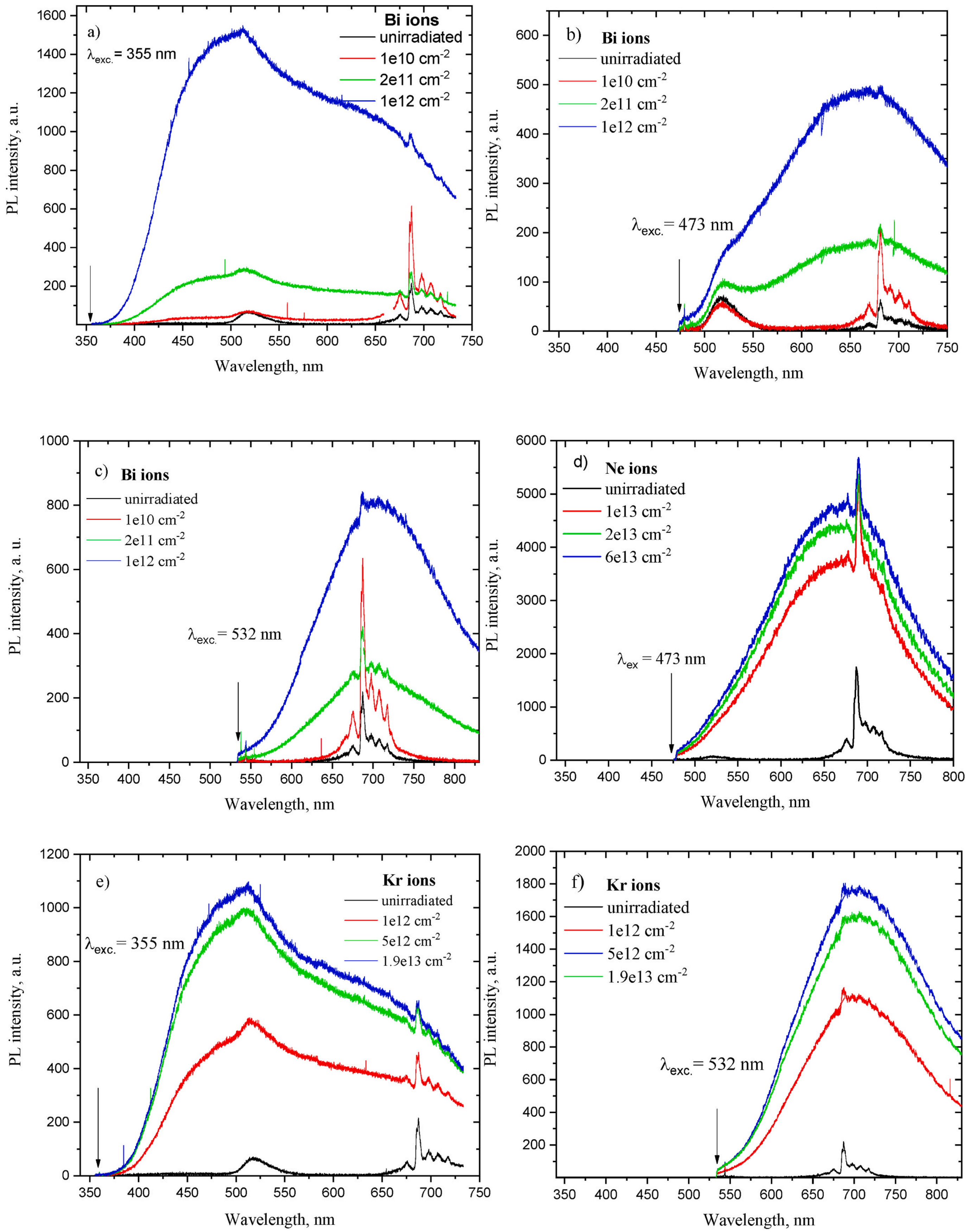

Fig. 3. PL spectra from $MgAl_2O_4$ irradiated with 670 MeV Bi (a,b,c), 23 MeV Ne (d) and 100 MeV Kr (e,f) ions. The spectra were corrected for system response. Positions of the excitation wavelength are shown by arrows.

shallow and deep defects, within the band gap of the materials. It was concluded also that at excitation wavelengths higher than 300 nm, the electrons trapped at shallow states were detrapped followed by re-trapping in the deep states [35].

In general, the above papers [34,35] show very complex situation with impurity and defect related luminescence in $MgAl_2O_4$. The situation becomes even more complicated knowing that SHIs may produce the widest possible spectrum of structural imperfections in spinel via relaxation of dense electronic excitations as well as via elastic collisions – from extended defects, latent tracks, to point defects and their complexes in different charge states. Since the Bi ion tracks retain the crystal structure, we can assume that they also have the same structure as the tracks of 350 MeV Au and 200 MeV Xe ions– defective core region (~2 nm) surrounded by disordered region with 5–6 nm in radius [7,8]. It was found recently that the PL intensity of the 650 nm band in 152 MeV Xe ion irradiated spinel has a maximum at fluence $\Phi \sim 10^{13}$ cm^{-2} when track cores start to overlap [17]. This means that luminescent centers, whose nature must still be determined, are produced and accumulated in disordered regions around track cores and their multiple overlapping, up to ten times at 10^{13} cm^{-2}, results in the increase of the luminescence yield.

Identification of radiation defects associated with obtained PL spectra is significantly hampered by the absence of prominent bands in absorption spectra in SHI irradiated spinel at wavelengths higher 300 nm [10,11,14]. Very recent measurements of excitation spectra of 1.7 eV PL band in $MgAl_2O_4$ irradiated with 220 MeV Xe ions have revealed two bands with maxima at 3.4 eV (365 nm) and 2.7 eV (459 nm) apart the well-known band attributed to F^+-centers in UV region [14]. It was suggested that emission in the 1.7 eV band may be due to intra-center transitions in centers with energy levels around 3.4 eV and not yet attributed to any known defects. It is important to note that the above measurements were carried out in non-confocal geometry and provided information from the entire irradiated layer. According to Ref. [35], the luminescence registered during excitation by photons with $\lambda > 300$ nm is related to the release of electrons from shallow traps and re-trapping in deep states followed with radiative decay. Strong cation disorder induced by swift heavy ions and revealed using transmission electron microscopy as well as by Raman spectroscopy suggests the formation of a high concentration of shallow traps. In general, ion tracks may be considered as regions with large variation of the Mg/Al ratio from the ideal ratio, i.e. as a mixture of normal and inverse spinel. To some extent, this is a unique situation not realized with other types of ionizing radiation. According to Ref. [35], the band gap width of inverse spinel is 4.95 eV, which is 1.05 eV lower compared to the defect free normal spinel. This may increase the probability of the charge transfer from shallow to deep states in inverse spinel since the deep states of F^{2+}centers, for example, are found to be very close to the shallow states and almost merge with them, showing as a single broad band [35]. In particular, this may explain the reduction of characteristic radiative decay time in 1.9 eV PL band with increase of 152 MeV Xe ion fluence found in our previous work [17]. Also, the decay kinetics observed during photobleaching by excitation laser can be considered as confirmation of the release of electrons from shallow traps. Fig. 4 shows the time variation of the integral PL signal from two spectral regions during excitation of Bi ion irradiated spinel at 355 nm.

In summary, we can only state with confidence that the observed broad band photoluminescence is due to SHI related radiation defects and identification of its nature will directly depend on progress in determining the morphology of ion track regions in spinel and the mechanisms of charge carrier transport under conditions of high concentration of antisite defects with variable Mg/Al ratio.

4. Conclusions

It was found that photoluminescence spectra associated with SHI induced radiation defects in the near surface layer of $MgAl_2O_4$ single crystals are broad overlapping bands lying in the spectral range from the excitation wavelength to at least 850 nm. Such spectral composition is attributed to a large variation of the Mg/Al ratio within the ion track regions that retain a crystalline structure even at ultrahigh values of the electronic stopping power 36.6 keV/nm.

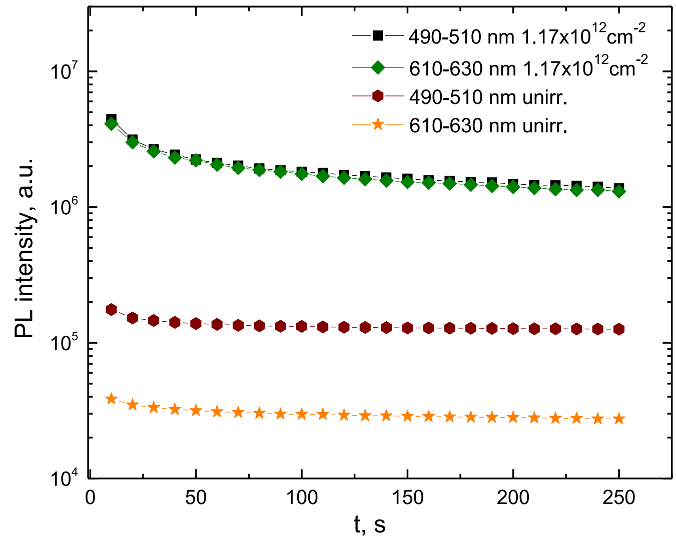

Fig. 4. Integral PL intensity versus photoexcitation time measured from intact and Bi ion irradiated $MgAl_2O_4$. Ion fluence 1.17×10^{12} cm^{-2}.

CRediT authorship contribution statement

V. Skuratov: Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis, Conceptualization. **O. Korolik:** Resources, Methodology, Data curation. **M. Mamatova:** Visualization, Software, Investigation, Formal analysis, Conceptualization. **J. O'Connell:** Writing – review & editing, Visualization, Software, Resources, Methodology, Data curation. **N. Kirilkin:** Methodology, Formal analysis, Data curation. **A. Dauletbekova:** Supervision, Resources, Investigation, Conceptualization. **A. Akilbekov:** Validation, Supervision, Project administration, Investigation.

Declaration of competing interest

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

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Data availability

Data will be made available on request.

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