

# Accumulation of radiation defects and modification of micromechanical properties under MgO crystal irradiation with swift $^{132}\text{Xe}$ ions

G. Baubekova<sup>a,b</sup>, A. Akilbekov<sup>a</sup>, E. Feldbach<sup>b</sup>, R. Grants<sup>c</sup>, I. Manika<sup>c</sup>, A.I. Popov<sup>c</sup>, K. Schwartz<sup>d</sup>,  
E. Vasil'chenko<sup>b,c</sup>, M. Zdorovets<sup>a</sup>, A. Lushchik<sup>b</sup>,

<sup>a</sup>L.N. Gumilyov Eurasian National University, Munitpassov Str. 5, 0100008 Nur-Sultan, Kazakhstan

<sup>b</sup>Institute of Physics, University of Tartu, W. Ostwald Str. 1, 50411 Tartu, Estonia

<sup>c</sup>Institute of Solid State Physics, University of Latvia, Kengaraga 8, Riga LV-1063, Latvia

<sup>d</sup>GSI Helmholtzzentrum für Schwerionenforschung, Planckstr. 1, 64291 Darmstadt, Germany

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## ABSTRACT

Accumulation of  $F$ -type defects under irradiation of MgO crystals by 0.23-GeV  $^{132}\text{Xe}$  ions with fluence varying by three orders of magnitude has been investigated via the spectra of optical absorption and low-temperature cathodoluminescence. The number of single centers continuously increases with fluence without any marks of saturation. At the highest fluence, a mean volume concentration of  $3.1 \times 10^{19}$  and  $3.35 \times 10^{19} \text{ cm}^{-3}$  is reached for  $F$  and  $F^+$  centers, respectively. The  $F^+$  emission strongly dominates in the cathodoluminescence of irradiated MgO and its enhancement with fluence is detected. However, the creation efficiency of the  $F_2$  aggregate centers is very low and fluence dependence has a complicated shape. Radiation-induced changes of micro-mechanical properties of the same samples have been analysed; the depth profiles of hardening correlate with the ion energy loss. A joint contribution of ionization and impact mechanisms in the formation of structural defects under MgO irradiation with Xe ions is considered.

## 1. Introduction

Magnesium oxide is a fascinating material exploited for various applications in science and technology (see [1] and references therein). In particular, MgO crystals exhibit very high tolerance to intentional irradiation or prolonged stay in harsh radiation environment. Therefore, MgO crystals and ceramics have potential in nuclear applications such as nuclear fuels, waste storage, fission reactor materials and even are considered as promising materials for diagnostics/windows in future deuterium-tritium fusion devices (see, e.g., [2–4]). For nuclear applications, the basic criteria are structural and mechanical integrity as well as rather low efficiency of structural defect accumulation under certain conditions of irradiation (absorbed energy, temperature, etc.).

In general, functionality of many materials is limited by insufficient radiation tolerance, which strongly depends on the accumulation of stable (long-lived) structural defects. That is why the understanding of the mechanisms responsible for radiation-induced damage in materials of different type as well as the prospects to control/decrease/suppress this damage are of crucial importance. There are many experimental studies devoted to the structural defects (Frenkel and extended ones) as well as to the processes of their creation and thermal annealing in MgO

irradiated with fast fission neutrons, swift ions or high-energy electrons (see [1,2,5–20] and references therein). In addition, several theoretical studies on kinetics of intrinsic defects and clusters in MgO were also performed [15,21–24].

It is generally accepted that the universal knock-on mechanism of radiation damage connected with the elastic collisions of incident particles with atoms/nuclei of metals and alloys is the dominant one in case of fast neutrons [25]. On the other hand, the creation of structural defects in a number of wide-gap materials (WGMs), in particular, in the majority of alkali halides is determined by the decay/recombination of radiation-induced electronic excitations (EEs) (see, e.g., recent review [26]). However, these EE-related mechanisms are realized only in WGMs, where the formation energy of Frenkel defects (interstitial-vacancy pairs) does not exceed the energy gap,  $E_{\text{FD}} < E_g$ .

At the same time, the opposite inequality,  $E_{\text{FD}} > E_g$  is valid for binary and complex wide-gap metal oxides, where the impact (collision) mechanism long time was considered solely responsible for radiation damage. In case of WGM irradiation with ~GeV swift heavy ions (SHIs), which provide extremely high density of EEs within cylindrical ion tracks ( $LET > 30 \text{ keV/nm}$  [27,28]), the situation is more complicated. There are clear manifestations that ionization losses (via

Corresponding author.

E-mail address: [aleksandr.lushchik@ut.ee](mailto:aleksandr.lushchik@ut.ee) (A. Lushchik).

complicated EE-related mechanisms) can also contribute to the creation of structural defects by SHIs even in WGMs with  $E_{FD} > E_g$  [28–33]. Note that one of such mechanisms, hot electron-hole recombination (before total relaxation of carriers within conduction and valence bands) could be attenuated to some extent by targeted doping of WGMs with luminescent impurities [20,29,32–34]. Still the elucidation of a real input of the ionization mechanisms in structural damage under WGM irradiation with SHIs is far from being completed.

In the present paper, a special emphasis is given to the accumulation of  $F$ -type Frenkel defects (via optical absorption) as well as to the corresponding changes in the spectra of cathodoluminescence and micro-mechanical properties of highly pure MgO single crystals under their irradiation by 0.23 GeV  $^{132}\text{Xe}$  ions with fluence varying by nearly three orders of magnitude.

## 2. Experimental

Highly pure MgO single crystals were grown at the Institute of Physics (Tartu, Estonia) by a variation of arc fusion technique with three recrystallization cycles. The concentration of Fe ions, the main impurity in the crystal, was up to 3 ppm. The samples with dimensions of  $\sim 5 \times 5 \times 1 \text{ mm}^3$  were irradiated at room temperature (RT) by 0.23 GeV  $^{132}\text{Xe}$  ions (1.75 MeV/u, current density  $10 \text{ nA/cm}^2$ ) with varying fluence of  $\Phi = 5 \times 10^{11} - 3.3 \times 10^{14} \text{ ions/cm}^2$  at the DC-60 cyclotron in Astana, Kazakhstan. According to SRIM calculations [35], the range of  $^{132}\text{Xe}$  ions with the used energy in MgO was about  $R \approx 14 \mu\text{m}$ .

The spectra of optical absorption were measured at RT using a high-absorbance spectrometer JASCO V-660 with a double monochromator (1.5–6.5 eV), while a homemade setup based on a vacuum monochromator VMR-2 and the hydrogen discharge source allowed measurements in vacuum ultraviolet (VUV, up to 10 eV) region as well. In the latter case, the number of incident photons from the hydrogen discharge in a flow capillary tube was maintained constant by varying the monochromator slit width and using the constant signal from sodium salicylate for normalization. Note that the difference between two absorption spectra measured at RT before and after irradiation (i.e. for the virgin and the same sample after irradiation) is considered to be radiation-induced optical absorption (RIOA).

Cathodoluminescence (CL) spectra were measured at the sample excitation by an electron beam of 10 keV energy and 0.1  $\mu\text{A}$  current (for details see [36]). According to the CASINO simulation [37], the penetration depth of such electrons is about 0.5  $\mu\text{m}$ . The CL setup was equipped with a close cycle helium cryostat (5–400 K) and two monochromators covering a wide spectral range of 1.5–10 eV: an ARC SpectraPro 2300i monochromator and a homemade vacuum double monochromator. To avoid crystal surface charging during electron beam excitation, the 3-nm Pt films were deposited on all samples.

Radiation-induced changes of micro-mechanical properties were characterized by the Nanoindenter G200 (Agilent, USA) equipped with Berkovich and Vickers diamond tips. The measurements of hardness depth profiles were performed on the surfaces prepared by cleaving the samples along the direction of ion beam at a constant indentation depth (150 nm) ensuring small indents and a reasonable amount of data points with a good statistics (for details see Ref. [38]).

## 3. Results and discussion

Fig. 1 presents a set of RIOA spectra measured for MgO single crystals irradiated by  $^{132}\text{Xe}$  ions with fluence varying by about three orders of magnitude. The spectra comprise several well-pronounced broad bands peaked around 5.0, 3.48 and 2.16 eV, which were earlier analysed in the literature for additively coloured or heavily irradiated MgO crystals (see [5–11] and references therein). The non-elementary band at  $\sim 5 \text{ eV}$  is an overlapping of two bands with the maxima at 5.03 and 4.92 eV ascribed to the so-called  $F$  and  $F^+$  centers (two or one

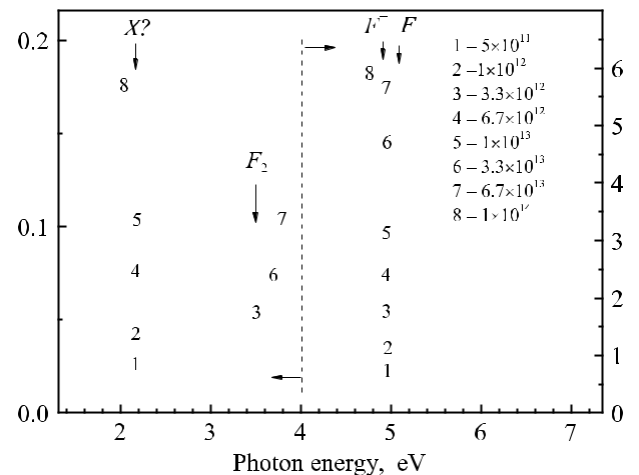


Fig. 1. Spectra of RIOA for MgO single crystals irradiated by 0.23-GeV  $^{132}\text{Xe}$  ions with different fluence at RT. All spectra are measured at RT.

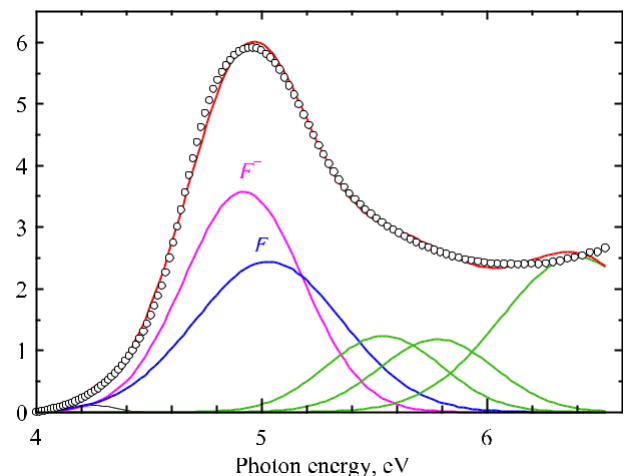
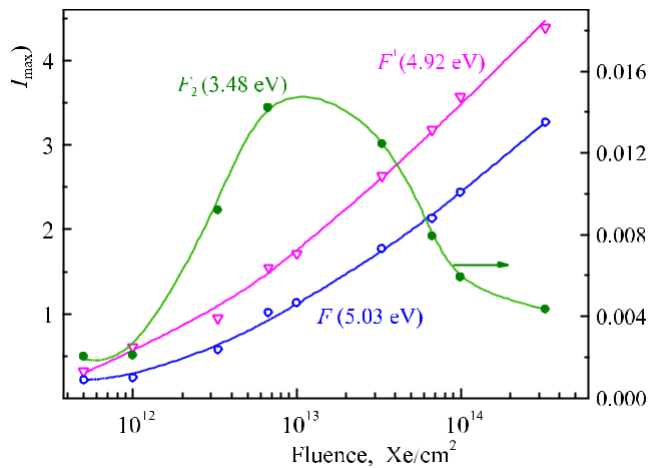


Fig. 2. Decomposition of the RIOA spectrum of MgO ( $\Phi = 1 \times 10^{14} \text{ Xe/cm}^2$ ) into Gaussian components. Symbols represent experimental curve.

electron trapped by an oxygen vacancy, respectively), the simplest aggregate  $F_2$  centers (two spatially close  $F$  centers) are responsible for the band peaked at 3.48 eV, while a complex band peaked at  $\sim 2.16 \text{ eV}$  belongs to the structural defects of still unclear origin. The iron-impurity-related bands are peaked at 4.26 and 5.74 eV. In case of high fluence (i.e. when the measured optical density exceeds 4) the top of complex UV band underwent saturation and was restored using the shape of the band at a measurable fluence.

It should be noted that the evolution of the RIOA at  $\sim 5 \text{ eV}$ ; 2.16 eV as well as around 5.8 eV (overlapping of Fe-related absorption and some unknown structural defects, see also Figs. 1 and 2) with the preheating temperature during annealing procedure for Xe-irradiated samples has been thoroughly analysed and the corresponding results will be presented in separate papers.

In order to analyse the fluence dependence of the number of  $F$ -type radiation defects, one of the main goals of the present paper, all nine RIOA spectra measured after MgO irradiation by SHIs with varying  $\Phi$  were decomposed into several Gaussian components. As an example, Fig. 2 depicts the decomposition results in the region of the 5-eV band for the MgO sample irradiated with  $\Phi = 1 \times 10^{14} \text{ Xe/cm}^2$ . The peak intensity  $I_{\text{max}}$  of the corresponding Gaussian or its area (integral)  $S$  as well as the RIOA values measured without decomposition at 4.92 and 5.03 eV (i.e. at the location of Gaussian maxima) can be taken as a measure of the number of  $F^+$  and  $F$  centers. All three versions of the defect number estimation give practically identical fluence dependence



**Fig. 3.** Dependence of the number of  $F$ -type centers (via the corresponding Gaussian maximum of RIOA) on ion-irradiation fluence.

that is important from a methodological point of view.

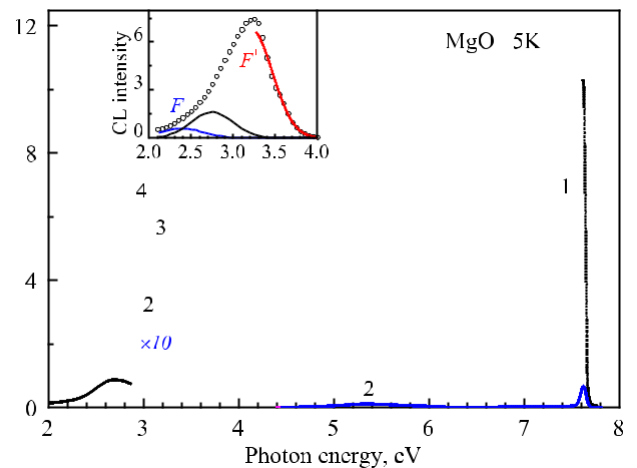
Fig. 3 shows the dependence of the number of  $F^+$  and  $F$  centers (via values of  $I_{\max}$ ) on the ion fluence. The number of  $F^+$  and  $F$  centers continuously increases with fluence up to  $\Phi = 3.3 \times 10^{14}$  ions/cm<sup>2</sup> (our experimental limit) without obvious marks of saturation. The dose dependence contains an approximately linear stage above  $\Phi = 3.3 \times 10^{13}$  Xe/cm<sup>2</sup> (the stage is more obvious in case of the linear abscissa scale), while significantly higher rate of defect accumulation is typical of lower irradiation fluences. It is generally accepted (see, e.g., [39,40]) that a fast growing nonlinear component of dose dependence below  $\Phi = 3.3 \times 10^{13}$  Xe/cm<sup>2</sup> can be explained by a partial creation of  $F$ -type centers on the basis of already existing in the sample as-grown oxygen vacancies. At the same time, the second nonlinear stage of defect accumulation (saturation) due to the possible aggregation of single  $F$ -type centers (for instance, formation of  $F_2$  and more complex defects) is not reached even at the highest fluence of  $\Phi = 3.3 \times 10^{14}$  ions/cm<sup>2</sup>.

The latter fact is also confirmed by the dependence of  $F_2$ -center number on irradiation fluence. According to Fig. 3, xenon ions create very low concentration of  $F_2$  centers (see optical density values at right ordinate) and, moreover, there is even decrease of the number of  $F_2$  centers (as well as  $F_2/F$  ratio) at  $\Phi \geq 10^{13}$  Xe/cm<sup>2</sup>. This result contradicts the case of LiF crystals, where comparable amount of  $F$  and  $F_2$  centers and nearly constant ratio of  $F_2/F$  was detected under swift-ion-irradiation up to saturation level different for N and Kr ions, when creation of more complex aggregates  $F_n$  became dominant [41,42]. Such difference in accumulation processes of  $F$ -type centers in MgO ( $E_{FD} > E_g$ ) and LiF ( $E_{FD} < E_g$ ) can be explained by different contribution of ionization and displacement mechanisms of radiation damage. It seems that collisions are a dominant way of oxygen vacancy creation in MgO (see also volume concentration profile of  $F$ -type centers in Refs. [8,9]), while highly efficient EE-related mechanisms (ionization loss) in LiF are preferable for  $F_n$  creation. Note that significantly higher value of  $F_2/F$  absorption ratio was detected in neutron-irradiated MgO with respect to that in  $\sim$ GeV <sup>238</sup>U-irradiated sample [30].

The concentration of  $F$ -type defects induced in MgO by SHI irradiation can be estimated by means of the well-known Smakula-Dexter formula [5,43]. In case of SHI irradiation with the value of ion range  $R$  significantly smaller than a sample thickness, the areal density of defects equals

$$n [\text{cm}^{-2}] = k \times 10^{15} \times 2.3 I_{\max}$$

where  $I_{\max}$  is the radiation-induced optical density at the maximum of a certain absorption band and a coefficient  $k$  takes into account the oscillator strength  $f$  and a FWHM for  $F$ -type center absorption band as well as refraction index for MgO (1.47). Considering  $f = 0.8$  for both  $F$  and



**Fig. 4.** Cathodoluminescence spectra measured for virgin (curve 1) and ion-irradiated (curves 2–4) MgO crystals under excitation by 10-keV electrons at 5 K. 2 –  $\Phi = 5 \times 10^{11}$  Xe/cm<sup>2</sup>; 3 –  $\Phi = 1 \times 10^{12}$  Xe/cm<sup>2</sup>; and 4 –  $\Phi = 1 \times 10^{13}$  Xe/cm<sup>2</sup>. Inset shows the decomposition of  $\sim 3.1$  eV band (symbols – experimental curve) into Gaussians for the MgO crystal irradiated with  $\Phi = 1 \times 10^{13}$  Xe/cm<sup>2</sup>. Curve 2 is multiplied by factor of 10.

$F^+$  centers and based on our decomposition results, FWHM ( $F$ ) = 0.77 eV, FWHM( $F^+$ ) = 0.62 eV, we obtain the values  $k$  as 5.76 and 4.64 for  $F$  and  $F^+$  centers, respectively. The mean volume concentration  $N$  [cm<sup>-3</sup>] can be calculated via  $N = n/R$ , that makes  $N(F) = 1.1 \times 10^{19}$  cm<sup>-3</sup> and  $N(F^+) = 1.3 \times 10^{19}$  cm<sup>-3</sup> for  $\Phi = 1 \times 10^{13}$  Xe/cm<sup>2</sup>, while  $N(F) = 2.3 \times 10^{19}$  cm<sup>-3</sup> and  $N(F^+) = 2.7 \times 10^{19}$  cm<sup>-3</sup> at the fluence of  $\Phi = 1 \times 10^{14}$  Xe cm<sup>-2</sup>.

Fig. 4 shows the CL spectra measured under 10-keV electron excitation at 5 K for virgin and ion-irradiated MgO single crystals. For a virgin sample, the spectrum contains a narrow peak at  $\sim 7.6$  eV, a complex broad band peaked at 5.3 and a weak complex CL at 2.3–3.3 eV. According to the literature, a single unresolved peak at  $\sim 7.6$  eV consists of a narrow line of the resonant emission of Wannier type free excitons and the emissions of several bound excitons connected with different impurity ions/defects [44,45]; the broad band at 5.3 eV is considered as radiative electron recombination at trapped-hole centers (a hole at oxygen near certain impurities or as-grown defects, i.e. bivalancies), while CL in a visible region is usually ascribed to the recombination of mobile holes with the electrons localized at as-grown-defects/impurities (see [17,18] and references therein).

The irradiation of MgO with Xe-ions causes drastic changes in CL spectra (curves 2–4 in Fig. 4). The sharp attenuation of exciton-type emissions with ion fluence is due to the appearance of radiation-induced structural defects where efficient non-radiative decay of mobile excitons takes place, while the suppression of CL at  $\sim 5.3$  eV can be explained by the reabsorption of this emission by  $F$  and  $F^+$  centers created by SHI-irradiation. On the other hand, the transformation of visible CL and significant enhancement of the luminescence peaked at  $\sim 3.1$  eV is also connected with radiation-induced structural defects. The decomposition of this CL band in the irradiated sample (see inset in Fig. 4) gives roughly three Gaussian components related to the emission of  $F$  and  $F^+$  centers (peaks at 2.4 and 3.2 eV with a FWHM = 0.6 eV for both components [7,10]) and a band at  $\sim 2.8$  eV tentatively ascribed to SHI-induced stress/bivalancies [18,31,32,46]. Note that according to the literature data [31,32,47], the  $F/F^+$  emission ratio depends on the type of external influence (fast neutrons, SHIs, thermochemical reduction) on MgO crystals. In Xe-irradiated crystals (similar to the irradiation with  $\sim$ GeV uranium ions and contrary to the case of fast neutrons [32]), the emission of  $F^+$  centers is a dominant one.

According to Fig. 4 there is an obvious enhancement of  $F^+$  and  $F$  emission with xenon fluence and the intensity of these emissions can be even used as a measure of radiation-induced defects. Nevertheless, it

should be taken into account that the emissions of  $F$  and  $F^+$  centers under electron excitation could arise both at the direct excitation of the corresponding centers or after secondary processes involving the transformation of the radiation-induced centers (in our case  $F$ ,  $F^+$  and empty oxygen vacancies created by SHIs) by numerous electron-hole ( $e-h$ ) pairs formed by an electron beam under measuring the CL spectra (see, e.g., [31,36,47]). Only in the case of direct defect excitation the  $F$  (or  $F^+$ ) emission intensity is proportional to the number of radiation-induced  $F$  ( $F^+$ ) centers. To exclude possible secondary reactions – in particular,  $F$  emission can arise at the recombination of conduction electrons (from  $e-h$  pairs formed by an electron beam) with  $F^+$  centers; or the  $F^+$  emission could be the result of a hole recombination with a radiation-induced  $F$  center – the  $F/F^+$  ratio in the CL spectrum should be compared with that obtained from the RIOA spectra. At the same time, just the difference of  $F/F^+$  ratio in the CL spectra for MgO crystals that undergone additive coloration or irradiated with fast neutrons and SHIs allowed to suggest the contribution of both EE-related and collision mechanisms to radiation damage under WGM irradiation by SHIs [31,32,47].

In addition, a significant attenuation of a spectrally integrated CL has been recently detected in many WGMs exposed to irradiation with fast neutrons or swift ions [36,40,48,49]. Besides simple emission reabsorption by radiation-induced structural defects, this effect can be partly connected with radiation-induced strengthening of non-radiative processes under harsh radiation conditions. The detailed study of these processes still lies ahead.

Fig. 5 illustrates the variation of hardness and SRIM-calculated energy loss along the Xe-ion path in MgO irradiated with different fluences. The ion-induced hardening becomes detectable at  $\Phi > 10^{11}$  Xe/cm<sup>2</sup> corresponding to a stage of track overlapping. The hardness increases with fluence and at  $\Phi = 10^{14}$  Xe/cm<sup>2</sup> turns to saturation at about 15.6 GPa (hardening effect  $\Delta H/H_0 \sim 50\%$ ).

The hardness variation in the initial part of ion range correlates with the depth behaviour of electronic energy loss. It should be taken into account that in the incoming part of Xe ion range (up to 5  $\mu$ m) the electronic stopping power exceeds the 22 keV/nm threshold for the formation of latent tracks and creates ultimate excitation [50]. An exception is the end-of-range region where the hardness at high fluence displays a maximum. In this region, the electronic stopping power of ions decreases to small values, while the nuclear stopping reaches the maximum and dominates in the damage creation. Ion-induced extended defects, including dislocations and complex defects are considered to be the main cause of hardening [38]. None or small contribution of the nuclear mechanism to hardening is observed at  $\Phi < 10^{12}$  Xe/cm<sup>2</sup>.

The comparison of ion-induced hardening data for the crystals of

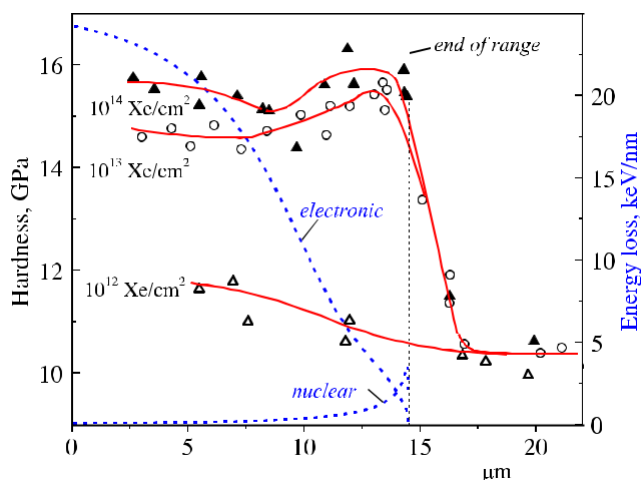


Fig. 5. Variation of hardness and energy loss along the path of 0.230 GeV <sup>132</sup>Xe ions in MgO crystals at different fluence.

different purity level shows a similar effect both for highly pure MgO single crystals (hardening effect  $\sim 45\%$ ) and commercial crystals from different suppliers (e.g., samples from MTI Company, USA with purity of 99,85 and main impurities of Ca (0.13%), Fe and Cr) irradiated with 150 MeV Kr ions (hardening effect  $\sim 40\%$ ) [38].

#### 4. Concluding remarks

In highly pure MgO single crystals, the number of radiation-induced  $F$  and  $F^+$  centers determined via the spectra of radiation-induced optical absorption continuously increases in two stages with different accumulation rates at the rise of fluence of 0.23 GeV Xe ions from  $5 \times 10^{11}$  up to  $3.3 \times 10^{14}$  ions/cm<sup>2</sup>. The linear stage representing mainly the radiation creation of novel oxygen vacancies starts at about  $\Phi = 3.3 \times 10^{13}$  Xe cm<sup>-3</sup> and continues without any marks of saturation up to the highest available fluence of  $\Phi = 3.3 \times 10^{14}$  Xe cm<sup>-3</sup> that provides a mean volume concentration of  $3.1 \times 10^{19}$  cm<sup>-3</sup> and  $3.35 \times 10^{19}$  cm<sup>-3</sup> for  $F$  and  $F^+$  centers, respectively. On the other hand, the creation efficiency of the simplest aggregate  $F_2$  centers is very low and demonstrates fluence dependence of complicated shape. The latter fact testifies to rather low (but still measurable, see below) involvement of ionization mechanisms (EE-related), responsible for efficient creation of aggregate centers at higher fluence in wide-gap materials with  $E_{FD} < E_g$  (in particular LiF [41,42]), in damage of MgO ( $E_{FD} > E_g$ ) under swift ion irradiation.

The rise of the number  $F$  and  $F^+$  centers can be also estimated in MgO via the enhancement of the visible cathodoluminescence around  $\sim 3.1$  eV with Xe fluence. At the same time, drastic attenuation/suppression of cathodoluminescence takes place in other spectral regions due to the partial reabsorption by radiation-induced structural defects (incl.  $F$  and  $F^+$  centers) as well as tentative strengthening of non-radiative processes under Xe-ion irradiation. Note that the  $F/F^+$  number ratio values in MgO, which can be determined via the spectra of cathodoluminescence or radiation-induced optical absorption, are very different:  $F^+$  emission strongly dominates in the cathodoluminescence spectra (similar to MgO crystals irradiated by  $\sim$ GeV uranium ions [32]), while volume concentration of  $F$  and  $F^+$  centers determined via absorption spectra is approximately the same.

Secondary reactions of  $e-h$  pairs formed at the excitation of SHI-irradiated MgO crystals by an electron beam with radiation-induced  $F$  and  $F^+$  centers could misrepresent the real ratio of center numbers estimated via cathodoluminescence spectra. It has been already mentioned that the  $F/F^+$  emission ratio also depends on the type of external expose (fast neutrons, SHIs, thermochemical reduction [31,32,47]) on MgO crystals. Such difference testifies to the non-identity of the creation mechanisms of  $F$ -type centers by swift heavy ions and the collision mechanism typical of the material irradiation with fast fission neutrons. In addition, the depth profiles of hardening in MgO crystals correlate with the energy loss of Xe ions and also confirm a joint contribution of ionization and elastic collision mechanisms in the formation of extended defects (see also Refs. [33,34]).

#### 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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