



# Luminescent and dosimetric properties of ultrafine magnesium oxide ceramics after high dose irradiation



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## HIGHLIGHTS

- Ultrafine anion-defective MgO ceramics are synthesized.
- Luminescence of F-type centers increases with a growth of synthesis temperature.
- TL of deep traps is due to tunneling recombination of charge carriers.
- TL dose response goes sublinearly at doses 1.5–80 kGy of a pulsed electron beam.

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## ABSTRACT

The luminescent and dosimetric properties of ultrafine MgO ceramics synthesized in strongly reducing conditions at  $T = 1100\text{--}1400\text{ }^\circ\text{C}$  are investigated. The growth of photo- and cathodoluminescence output at 2.0–3.5 eV (400–600 nm) is found. It is due to an increase in concentration of single oxygen vacancies and their aggregates. It was established that thermal treatment leads to TL intensity growth after high dose irradiation of the samples by a pulsed electron beam (130 keV). The tunneling mechanism of charge carriers' recombination occurs after this treatment as well. The presence of tunneling recombination is proved by the analysis of the shape and temperature dependence of TL isothermal decay curves. The possibility of using synthesized ceramics for high dose TL dosimetry of ionizing radiations is shown.

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## 1. Introduction

Recently the intensive studies aimed at creation of high dose luminescent detectors of ionizing radiation are carried out. Such interest is due to wide application of this radiation in science and technology. In this case the absorbed dose can reach 1–100 kGy. The most successful experiments which confirm the possibility of high dose measurements were carried out by using the method of thermoluminescent (TL) dosimetry. Phosphors with low grain size are a new type of materials which are promising for high dose measurements due to their high radiation resistance (Kortov, 2010; Numan Salah, 2011).

Magnesium oxide (MgO) is a famous functional material which

is widely used in science and technology. Many papers (Las and Stoebe, 1984; Soliman, 2009; Kumar et al., 2011; Lushchik et al., 2012; Popov et al., 2013) are devoted to study of radiation-induced processes and luminescent properties of different MgO crystalline forms. A comparative investigation of luminescence in bulk and nanocrystalline MgO during excitation by synchrotron radiation was carried out (Popov et al., 2013). The blue shift in the excitation spectrum of 2.95 eV luminescent band associated with the quantum limit effect when the nanoparticle size decreases to 10–15 nm, was found. Other authors carried out the study of influence of sol–gel synthesis conditions of nanostructured MgO on the phase composition, grain size and photoluminescence (PL) (Kumar et al., 2011). The dependence of PL intensity on temperature and atmosphere during sample synthesis was established. A number of luminescent bands which are not observed earlier was revealed. These bands are presumably due to aggregates of oxygen vacancies and impurity defects. The TL and dosimetric properties of MgO micropowders after high dose gamma-irradiation were

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investigated (Soliman, 2009). However, the luminescent and dosimetric properties of MgO with low grain size after high dose irradiation are studied insufficiently.

It is known that phosphors with low particle size, in comparison with their bulk analogues, are characterized by lower TL output at small doses (Kortov, 2010; Numan Salah, 2011). In this regard, the problem of an increase in their TL sensitivity to radiation arises. One of the methods to increase TL output in wide-gap oxide phosphors is high temperature treatment in vacuum in reducing conditions. It is known that such treatment leads to an increase in concentration of F-type centers associated with oxygen vacancies. These centers determine luminescent properties of oxide materials (Akselrod et al., 1993; Monge et al., 2000).

The aim of the present work is to study the influence of high temperature treatment in reducing conditions on luminescent and dosimetric properties of MgO ceramics. The possibility of its application for high dose dosimetry of ionizing radiation is also assessed.

## 2. The samples and experimental techniques

MgO samples in the form of compacts (5 mm in diameter and 1 mm thick) were used as objects of the study. They were made from nanopowder by using the method of uniaxial cold pressing at unit pressure of 1000 kgf/cm<sup>2</sup>. The initial powder with grain size of 45–75 nm was made by plasma synthesis method (Vollath, 2008) (“Plasmotherm” Company, Russia). The MgO content was 99.8 wt.%, according to the data from the producer. The impurities of Fe<sub>2</sub>O<sub>3</sub> (no more than 0.1%) and SiO<sub>2</sub> (no more than 0.1%) are present in the powder under study. High temperature treatment (T = 1100–1400 °C, 3 h) of the compacts was carried out in a vacuum (10<sup>-3</sup> – 10<sup>-4</sup> Torr) furnace in the presence of carbon (graphite rod) to create strongly reducing conditions. It is known that the size of nanoparticles increases during high temperature treatment of nanopowders and compacts. The influence of synthesis conditions on the structure and grain size of the samples under study would require a separate and more detailed study. A preliminary assessment of particle size with a scanning electron microscope (SIGMA VP, Carl Zeiss, Germany) was carried out. It shows that the mean grain size of MgO ceramics synthesized at high temperature ranged in 250–500 nm. Thus, the samples under study were ultrafine ceramics.

The samples were irradiated by a pulsed electron beam of an accelerator (the pulse duration is 2 ns, the electron energy is (130 ± 1) keV, the current density is 60 A/cm<sup>2</sup>) at room temperature for TL and pulse cathodoluminescence (PCL) excitation. The absorbed dose from one pulse was experimentally determined by using a film dosimeter PD(F)R-5/50. The value of 1.5 kGy/pulse was obtained (Afanas'ev et al., 2005). TL was measured during linear heating with the rate of 2 K/s by FEU-130 photomultiplier tube with the maximum of spectral sensitivity at 400–420 nm.

PCL was measured by « KLAVI » spectrometer (Russia) in the range of 300–800 nm. The uncertainty in wavelength measurement was ±0.75 nm. The spectral resolution was no more than 2 nm. The measurement was carried out by the registration of mean spectrum obtained by several (10–20) electron pulses with the frequency of 1 Hz.

A spectrometer LS-55 (Perkin Elmer, USA) was used for PL measurements. The xenon lamp with the 150 W power was as light source. It worked in pulse mode with the pulse frequency of 50 Hz. The uncertainty in wavelength determination was ±1 nm. The measurements were carried out in phosphorescence mode with integration time of 12.5 ms.

## 3. Results and discussion

### 3.1. Pulsed cathodoluminescence

The PCL spectra of the initial compacts and thermally treated ones in vacuum in reducing conditions at different temperatures are presented in Fig. 1. It is seen that the band at 1.65 eV (750 nm) dominates in the PCL spectrum of the initial samples. Weak emission at 2.0–3.5 eV (400–600 nm) is also observed. The PCL intensity at 1.65 eV band changes slightly with growing annealing temperature. In this case PCL at 2.0–3.5 eV increases by the order at T = 1400 °C.

According to the literature, luminescence of MgO at 2.0–3.5 eV is due to relaxation of F-type centers (Edel et al., 1979; Rosenblatt et al., 1989). Thus, F-centers (oxygen vacancies with two captured electrons) have an emission band at 2.5 eV (500 nm). F<sup>+</sup>-centers (oxygen vacancies with one captured electron) have a luminescent band near 3.1 eV (400 nm). There is an opinion that the luminescence of aggregate F<sub>2</sub>-type centers can be observed in the spectral region under study (Bolton et al., 1981).

The obtained results show that the PCL band at 1.65 eV cannot be due to oxygen vacancies, because its intensity practically does not change after the high temperature treatment in reducing conditions. It is believed that it can be associated with the relaxation of excited states of iron ions (Fe<sup>2+</sup>) (McKeever et al., 1995). The presence of the noticeable iron oxide impurity in the samples under study evidences in favor of this assumption. The presence of Cr<sup>3+</sup> ions in the samples can raise the PCL at 1.6–1.8 eV too (Shablonin et al., 2015).

### 3.2. Photoluminescence

The PL spectra of the MgO samples under study in dependence on thermal treatment conditions are shown in Fig. 2. PL was excited near the absorption bands of F- and F<sup>+</sup>-centers (5.0 eV, 250 nm) (Edel et al., 1979; Rosenblatt et al., 1989). We decomposed the spectra under study into Gaussians. The example of such decomposition for ceramics annealed at T = 1200 °C is present in Fig. 2 (inset). The emission bands at 2.5 and 3.1 eV associated with F- and F<sup>+</sup>-centers, respectively, were observed in all spectra. These bands increase as a result of thermal treatment. Moreover, the luminescent band at 2.9 eV (440 nm) is observed in spectra. Some authors (Bolton et al., 1981) attribute the appearance of this band to

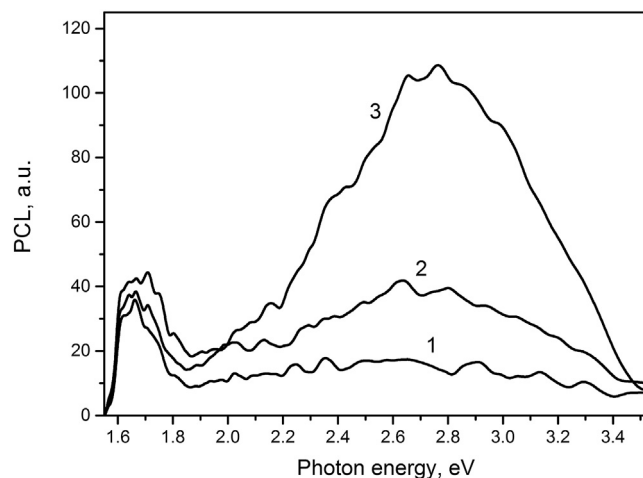


Fig. 1. PCL spectra of MgO: initial samples (1) and thermally treated ones at 1300 (2) and 1400 °C (3) for 3 h.

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