



Thermoluminescence and low-temperature luminescence of beryllium oxide



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HIGHLIGHTS

- We studied pristine and additively-colored BeO crystals and BeO-ceramics.
- We studied X-rays induced luminescence spectra at 6 and 293 K.
- We studied spectrally integrated thermoluminescence glow curves at 6–293 K.
- We determined parameters for dominate TL glow peak located below RT.
- A number of low-intensity TL peaks were found between 100 and 270K.

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ABSTRACT

Beryllium oxide in the forms of either single crystals (pristine, additively-colored) or hot-pressed ceramic samples was studied in the energy range of 1.2–6.2 eV using both the thermoluminescence (TL) and steady-state X-ray induced luminescence (XRL) techniques. The XRL emission spectra were recorded at 6 and 293 K, whereas TL glow curves were studied after X-ray exposure at $T_0 = 6$ K upon linear heating in the temperature range from 6 to 293 K. A search for TL manifestations of shallow trapping centers was carried out using a sensitive channel for TL registration in the range of more than six decades of change in intensity. The participation of shallow trapping centers in the process of recombination luminescence excitation at 6–293 K; branching electronic excitations between different recombination channels; the dominance of the self-trapped exciton and F-center emissions in spectra of the low-temperature recombination luminescence in BeO at 6–293 K were discussed.

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1. Introduction

Since the discovery of intense ultraviolet (UV) emission from beryllium oxide (Albrecht and Mandeville, 1954) and the development of the technology of growing large BeO single crystals (Austerman, 1963), BeO has been the subject of numerous studies. However, the number of research works devoted to luminescence and thermoluminescence (TL) of BeO single crystals at temperatures below 78 K remains relatively low. We are aware of several of them: Cooke et al. (1985, 1984); Petrenko et al. (2014); Shulgin et al. (1988) investigated both the luminescence and TL of BeO:Li, Na single crystals; Pustovarov et al. (2001) have studied relaxation of electronic excitations in BeO crystals using synchrotron radiation spectroscopy. All the other works cited for BeO single crystals, deal

with temperatures $T > 78$ K and are devoted to the study of scintillation (Ogorodnikov et al., 1996a; Shulgin et al., 1988) and dosimetric (Ogorodnikov et al., 1991b, 1996b) properties, as well as radiation processing (Belykh et al., 1997; Yakushev et al., 2000, 1999) and doping (Ogorodnikov et al., 1991a) of BeO single crystals. Also, there are several review papers on BeO single crystals (Kruzhalov et al., 1996; Ogorodnikov et al., 1994, 1995a; Ogorodnikov and Kruzhalov, 1997).

From the publications cited above, it follows that self-trapped excitons (STE) and F-type lattice defects are dominant luminescence centers in BeO. Pustovarov et al. (2001) have shown the possibility of excitation of the STE during recombination of zone electrons and holes assisted by different lattice defects. Recombination processes in BeO are well studied in the temperature range above 78 K. Elucidation of the role of shallow trapping centers in BeO requires the study of recombination processes at temperatures below 78 K. However, until now only fragmentary data for this

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temperature range are available. The possibility of excitation of STE and F-centers in recombination processes allowed us to use them as luminescence probes for studying manifestations of shallow trapping centers in BeO at temperatures of 6–293 K.

The aim of this research work is to study luminescence and thermoluminescence properties of undoped BeO in the forms of both the pristine and additively-colored single crystals in comparison with BeO-ceramics. The research work was carried out in the spectral (1.2–6.2 eV) and temperature (6–293 K) ranges using the methods of both the steady-state X-ray induced luminescence (XRL) and TL techniques.

2. Experimental details

The pristine beryllium oxide single crystals of optical quality were grown in the inverse temperature gradient from BeO-solution in a sodium tungstate melt (Maslov et al., 1980). The isomorphous capacity of BeO is fairly low. The Li^+ , Zn^{2+} , Mg^{2+} , B^{2+} , Al^{3+} impurities are the most common substitution ions in beryllium oxide. The background level of their concentration in the undoped crystals was 1–20 ppm. In this research we used only undoped both the pristine and additively-colored synthetic beryllium oxide single crystals. To obtain additively-colored crystals, pristine stoichiometric samples underwent thermal treatment in a metal beryllium vapor in order to produce anion nonstoichiometry (additive coloring). During three hours process the temperature level was 1900 °C and the beryllium vapor pressure value was 2.5 kPa (Gorbunov et al., 1987a, b). The concentration of anion vacancies (F-centers) were determined using an absorption spectroscopy of additively colored crystal in the absorption band at 6.6 eV (FWHM = 0.82 eV). Using Smakula's formula, the concentration value was estimated as $3 \times 10^{17} \text{ cm}^{-3}$. Samples of undoped BeO-ceramics were investigated in this case for comparison. We used samples of hot pressed (1900 °C) ceramics obtained from BeO-powder (Kiiko et al., 2003; Ogorodnikov et al., 1995b). Impurity content in the ceramic BeO-samples does not exceed 100–150 ppm.

All studies were performed in the laboratories at Ural Federal University. The steady-state X-ray-induced luminescence (XRL) spectra were recorded in the energy range of 1.2–6.2 eV at $T = 6$ and 293 K (vacuum better than 10^{-4} Pa). The thermoluminescence (TL) glow curves were recorded at spectral-integrated mode (200–650 nm) using a FEU-130 type photomultiplier tube. The BeO samples were irradiated by X-rays (Cu-anode, $U_a = 40$ kV, $I_a = 10$ mA) for 40 min in a vacuum at a temperature $T_0 = 6$ K. After exposure, the samples were kept at T_0 for a few minutes, and then heated at linear over the temperature range from 6 to 293 K (vacuum better than 10^{-4} Pa). All the spectra are normalized in intensity to one unit.

3. Results and discussion

Steady-state XRL spectra. Fig. 1 shows steady-state XRL spectra of three BeO samples recorded at temperatures $T = 6$ and 293 K. Intrinsic XRL-emission band at 4.9 eV dominates the luminescence spectrum in pristine BeO crystals, Fig. 1a. The wide UV-luminescence band in beryllium oxide single crystals has previously been described in terms of self-trapped exciton model (Pustovarov et al., 2001). According to this model, the broadband UV emission in BeO is due to radiative annihilation of self-trapped exciton, the hole core of which is a small-radius hole-polaron. Pustovarov et al. (2001) have shown that the excitation of the STE can occur not only during direct creation of a free exciton, but during recombination of zone electrons and holes assisted by different lattice defects. The recombination process is also

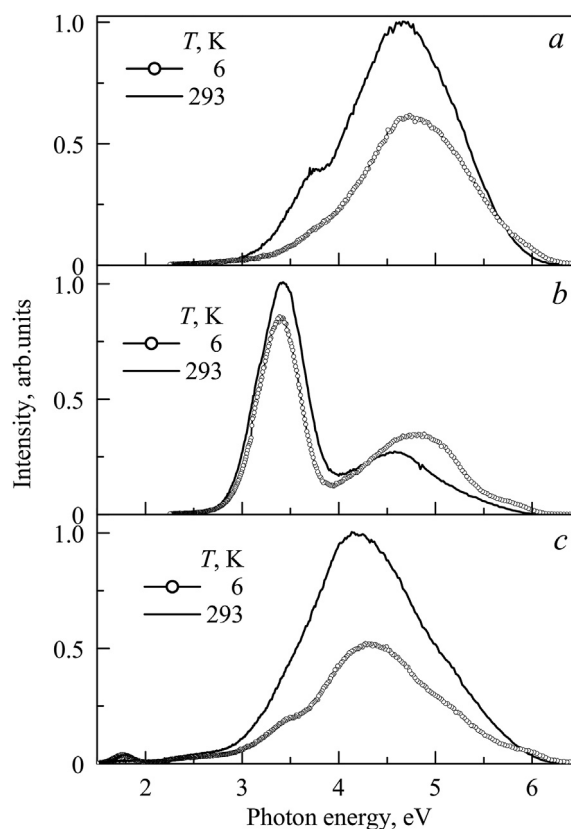


Fig. 1. Steady-state XRL spectra of beryllium oxide recorded at $T = 6$ and 293 K for pristine (a) and additively-colored (b) single crystals and ceramic samples (c).

accompanied by the creation of impurity-bound excitons, which relaxation contributes to the appearance of additional emission bands. This not only explains the appearance of various humps on the dominant XRL-emission band, but also explains an increase in the XRL-intensity when heated from 6 to 293 K, Fig. 1a. Additive coloring resulted in the appearance of new dominant XRL-emission band at 3.4 eV, Fig. 1b. Gorbunov et al. (1987b) have been investigated in detail the 3.4 eV-emission band in photoluminescence spectra of BeO single crystals in the temperature range above 78 K, and described it in terms of ${}^3A_1 \rightarrow {}^1A_1$ transitions in F-center centers. From Fig. 1b it follows that the XRL luminescence from F-centers in BeO is efficiently excited in recombination processes at $T = 6$ K. This is worth noting that during the heating from 6 to 293 K we can see a decrease in the intensity of UV emission band, but at the same time there is an increase in the intensity of the F-band at 3.4 eV. This may indirectly indicate a redistribution of electronic excitations between two excitation channels resulting to the relaxation of the electronic excitation responsible for the UV- and F-emission bands. The XRL-emission spectrum of ceramic samples shows a broad band with a maximum of about 4.4 eV. A large spectral width can testify to its complex origin. We can suggest that the observed XRL-spectrum includes apparently the two above mentioned bands at 3.4 and 4.9 eV, Fig. 1c. When heated from 6 to 293 K, the XRL-intensities in the neighborhood of 3.4 and 4.9 eV bands vary in different ways depending on the type of samples, Fig. 1. Low-intensity shoulder at about 3.9 eV in ceramic samples can be explained by the appearance of randomly distributed lattice defects formed during the synthesis of the BeO samples.

Briefly summarizing the results derived from the obtained XRL-data, we note the following: X-ray exposure leads to excitation of

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