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Nanostructured layers of anion-defective gamma–alumina – New perspective TL and OSL materials for skin dosimetry. Preliminary results

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HIGHLIGHTS

- Thin nanostructured layers (TNL) of Al_2O_3 with thickness of 5 mg/cm^2 were obtained.
- Its TL and OSL yields are related to contents of γ-phase and anion vacancies.
- Dose response of TNL shows linear behavior in a range of 10–5000 mGy.
- Luminescence properties of TNL are similar to such of anion-defective corundum.
- These properties are associated with centers of F-type.

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ABSTRACT

Thin- layer material based on nanostructured Al_2O_3 of the surface density 5 mg/cm^2 was obtained. The material is characterized by high OSL and TL yields comparable with those for TLD-500 which is one of the leaders among the TL and OSL detectors. The dose response, fading and dependence of TL yield on heating rate was studied. It is established that high luminescence yield of the samples under study correlates with the content of anion vacancies and γ-phase of Al_2O_3. The data for time-resolved luminescent spectroscopy are presented, which evidence for possible correlation between high TL and OSL activity and the F-type centers. It is noted that the material needs to be modified for successful use in dosimetry. In addition further studies to decrease the contribution of unstable (at 300 K) components to OSL and TL yields are required.

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1. Introduction

Current status of individual dosimetry control dictates active studies aimed at creation of high-performance storing luminescent detectors to measure the absorbed doses from β- and soft (≤30 keV) photon radiation in skin and eye lens (Blair et al., 2010; Rani and Sahare, 2013). This study is important because according to the requirements of the radiation safety regulations (ICRU-56, 1997; NRB-99/2009), surface density (ρ_s) of active layer of the detector must be sufficiently small and equal to the average ρ_s of the radiation-sensitive basal layer of skin, i.e. ~5–7 mg/cm^2. Then the required thickness for such thermoluminescent materials as LiF (detectors TLD-100, TLD-100H), α-Al_2O_3 (TLD-500), and $Li_2B_4O_7$ (TLD-800) is 10–30 μm. According to the standards (ICRU-56, 1997; NRB-99/2009) ρ_s of the cover (protective) layer of the detector must be equal to 5 mg/cm^2 and 40 mg/cm^2 for open skin and palm, respectively.

In spite of the requirements the detectors with $\rho_s \geq 10$ mg/cm^2 are still used in skin dosimetry (Mancosu et al., 2010; Pinto and Caldas, 2010). Therefore we estimated the measurement error of the doses absorbed by thick ($\rho_s \geq 10$ mg/cm^2) detectors compared to thin detectors ($\rho_s = 5$ mg/cm^2) under the protective layers of different thickness (Surdo A.I. et al., 2014). In particular, thick detectors with $\rho_s = 25$ mg/cm^2 understate the readings by no less than 25% for β- radiation with the maximum (in the spectrum) particle energy E_{β_max} within the range of 0.156–3.6 MeV, ρ_s of the protective layer being 5 mg/cm^2. The most dramatic increase of the

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relative error (from 40 to 80%) is observed with decreasing E_{β_max} from 1 down to 0.156 MeV.

Creation of detectors with ρ_s of the active layer 5 mg/cm^2 is associated with a number of problems, the main of which is small luminescent response and difficulty to obtain thin homogeneous active layers. Using nanotechnology we managed to obtain high-performance thin phosphor of the required thickness based on nanostructured Al_2O_3, the stored light sum of which may be measured by effects of thermally and optically stimulated luminescence (TL and OSL) (Surdo et al., 2013a).

The purpose of this study is to investigate the luminescent and dosimetric properties of the material and the dependence on crystalline and defective structure; and to elucidate the nature of the active luminescence centers responsible for the increased TL and OSL yields.

2. Experimental details

The thin nanostructured layers (TNL) of a 15–18 μm thickness and density of ~3 g/cm^3 (ρ_s = 4.5–5.4 mg/cm^2) were obtained by evaporation of a highly purified α-Al_2O_3 powder target using pulsed electron beam (E = 100 keV, τ = 20–300 μs, $j \geq 1$ MJ/cm^2), with subsequent sputtering on cooled aluminum, steel, and quartz glass substrates with the thickness of ~0.2–0.5 mm (Surdo et al., 2013a). According to TEM analysis the minimum, average and maximum sizes of the Al_2O_3 nanoparticles were 2, 5, and 10 nm, respectively (Sokovnin et al., 2013b). The TNL samples were irradiated with β-radiation of a ^{90}Sr/^{90}Y source (P = 0.5 mGy/s). The decay curves of continuous wave OSL and TL were investigated with an automatic instrument developed for this purpose. TL was recorded under the heating rate of 2 K/s. OSL was stimulated by white LED Kingbright KA-1010PW9AZC (Surdo et al., 2013b). The delivered irradiance to the sample is 4.1 mW/cm^2. The TL and OSL signals were recorded with FEU-142 photomultiplier using UFS-2 filter to separate the OSL signal from the stimulating light. In most cases, they were measured immediately after irradiation without taking the fading into account. Time-resolved spectra and kinetics of photoluminescence (PL) were studied using synchrotron radiation (SR) at DESY (Zimmerer, 1991). The TLD-500 moderate sensitivity detectors on the basis of a single crystalline anion-defective corundum (α- Al_2O_3) were used for comparative studies of luminescent and dosimetric properties.

3. Results and discussion

3.1. TL, OSL and other dosimetric properties

In Fig. 1 curves 1 and 2 show the TL curves for the obtained Al_2O_3 TNL samples deposited on steel and aluminum substrates. Independently of the substrate material both the curves display one complex peak of the same shape with a maximum at 450–460 K and a shoulder in the range of 365–370 K. Two luminescence bands with the maxima at 3.6 and 2.7 eV and half-width of 0.6 and 0.4 eV are observed in the TL spectrum, which are close to those for the luminescence of the F and F$^+$ centers (anion vacancies with two and one electrons, respectively) in α-Al_2O_3 (Evans et al., 1978; Surdo et al., 2001). The half-width of the TL peak at 455–460 K is ~2.5- to 3-fold greater than that of a TLD-500 detector based on anion-defective α- Al_2O_3 (curve 4). It is also seen that the TL yields normalized by mass are comparable for TLD-500 and TNL.

According to X-ray diffraction, differential scanning calorimetry and thermogravimetry analysis (XRD, DSC-TG) the samples under study contain mixture of the amorphous and γ-phases of Al_2O_3 (Sokovnin et al., 2013a). Concentration of the Al_2O_3 γ-phase increases with increasing annealing temperature (T_a) up to ~ 1000 K.

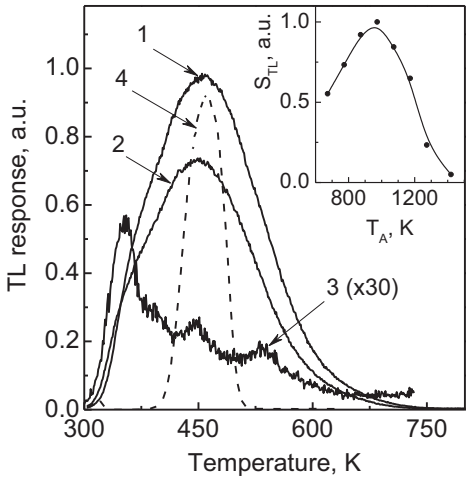

Fig. 1. Normalized by mass TL curves for the Al_2O_3 TNL samples on aluminum (1, T_a = 820 K), steel (2, T_a = 820 K; and 3, T_a = 1420 K) substrates and of TLD-500 detector (4) at D = 300 mGy. The inset shows $S_{TL}(T_a)$ for steel substrate.

When T_a reaches 1420 K the following sequence of phase transitions is observed: amorphous (520 K)→γ (1000 K)→ δ (1200 K)→θ (1250 K)→α (1420 K) (Sokovnin et al., 2013a). It is also important to note that an extremely low TL and OSL responses were found for amorphous TNL samples (Surdo et al., 2013a).

Therefore, we studied the TL curves and TL light sum (S_{TL}) for TNL as a function of T_a. It is seen (inset in Fig. 1) that with increasing T_a up to 1000 K the TL yield increases approximately twice. It is important that simultaneously the amorphous→γ-phase transition occurs. Further T_a increase up to 1420 K results in a significant fall of the TL yield. After annealing at 1420 K the TL curve (Fig. 1, curve 3) suffers essential changes, i.e. it shows three narrow low-intensity peaks at 350, 450, and 540 K. One of the main reasons for the observed decrease of the TL yield and drastic transformation of the TL curve after the annealing at 1420 K may be attributed to recovery of stoichiometric composition in the annealed TNL sample. According to DSC-TG and XRD data (Sokovnin et al., 2013a) mass of the samples increases at 870K $\leq T_a \leq$ 1420 K due to oxygen absorption from the air. Therefore, annealing at $T_a \geq$ 1000 K reduces not only the concentration of the γ-phase but also the concentration of anion vacancies, which determines luminescent response of the samples.

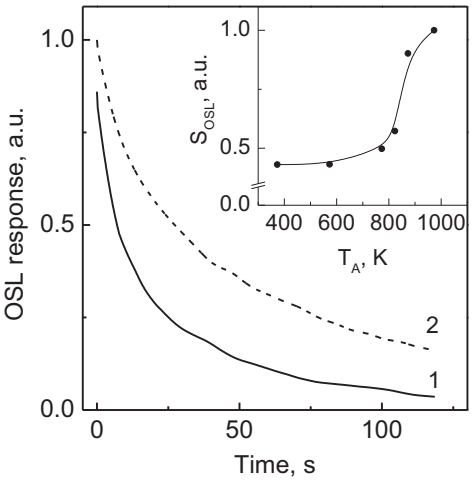

Fig. 2. Normalized by mass OSL curves for the γ-Al_2O_3 TNL sample (1, T_a = 970 K) and the TLD-500 detector (2) at 300 K. Inset shows $S_{OSL}(T_a)$.

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