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# The thermoluminescence and optically stimulated luminescence properties of Cr-doped alpha alumina transparent ceramics



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#### ABSTRACT

Polycrystalline  $Cr:\alpha-Al_2O_3$  transparent ceramics were fabricated by conventional solid-state processing under vacuum condition. The SEM microstructure photographs of  $Cr:\alpha-Al_2O_3$  transparent ceramics doped with different content of  $Cr_2O_3$  were investigated. The absorption, emission spectra, thermoluminescence and optical stimulated luminescence of  $Cr:\alpha-Al_2O_3$  transparent ceramics were comparable to those of  $Cr:\alpha-Al_2O_3$  crystals. The influence of different concentration of  $Cr_2O_3$  on the thermoluminescence and optical stimulated luminescence properties of  $Cr:\alpha-Al_2O_3$  transparent ceramics was discussed. It showed so interesting results with high TL sensitivity and high stability of OSL signal that  $Cr:\alpha-Al_2O_3$  transparent ceramics might be a promising material in TL dosimetry and replace  $Cr:\alpha-Al_2O_3$  crystals.

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#### 1. Introduction

The Thermoluminescence (TL) and optically stimulated luminescence (OSL) characteristics of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> single crystals has been the subject of considerable interest with increasing application in radiation dosimetry. TL or OSL is a luminescence phenomenon of an insulator or semiconductor which can be observed after being thermally or optically stimulated [1]. Several materials can be used as the thermoluminescence dosimetry, such as LiF [2], CaSO<sub>4</sub> [3], CaF<sub>2</sub> [4], Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub> [5], KSr<sub>4</sub> (BO<sub>3</sub>)<sub>3</sub> [6], CaAl<sub>2</sub>O<sub>4</sub> [7], and  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> [8].  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> was one of the earlier materials studied for its possible application as a radiation dosimeter well established in luminescence dosimetry [9].

With many favorable features such as superior mechanical strength, excellent chemical stability and low effective atomic number,  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> single crystals have many diversified applications [10–13] in laser host materials, radiation dosimeters, mechanical parts like wear surfaces, precision balls and insulations for nuclear reactor, etc.

 $\alpha\text{-Al}_2O_3$  doped with proper ions is a highly sensitive thermoluminescent for recording ionizing radiation. But, sustained efforts are still being made by either improving the TL/OSL properties of these materials by synthesizing them using different methods or by doping with different ions, such as C:Al<sub>2</sub>O<sub>3</sub> [14], Si, Ti:Al<sub>2</sub>O<sub>3</sub> [15] and Cr, Ni:Al<sub>2</sub>O<sub>3</sub> [16]. It has been shown that the luminescent centers play essential roles in the TL glow curves of  $\alpha\text{-Al}_2O_3$ . As luminescent centers, those dopants show interesting results in

dosimetry. And the efficiency of a phosphor material is often limited by the dynamics of the dopant ions which depend on crystal structure and ions concentration.

All the above reports were about  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> crystals. The use of Al<sub>2</sub>O<sub>3</sub> single crystals in dosimetry often require high temperature and sophisticated growing conditions, which raise their cost in comparison with transparent ceramics. Moreover, the content of dopant in crystals cannot be heavily doped. But the fabrication of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> transparent ceramics with nearly the same TL/OSL properties as those of single crystals do not require the above demanding conditions. And most importantly, they can be highly doped. Besides, large size, multi-layer active elements and multi-functional ceramics can be easily obtained. These advantages give much more freedom in TL/OSL designs. So it is a tendency that  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> transparent ceramics will replace  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> crystals in future.

However, there has been no report on the TL and OSL characteristics of  $Cr:\alpha$ -Al<sub>2</sub>O<sub>3</sub> transparent ceramics. In this paper, we present the influence of different concentration of  $Cr_2O_3$  on the TL and OSL properties of  $Cr:\alpha$ -Al<sub>2</sub>O<sub>3</sub> transparent ceramics.

#### 2. Materials and methods

High-purity  $Al_2O_3$  (99.95%),  $Cr_2O_3$  (99.99%) and  $La_2O_3$  (99.95%) powders were used to fabricate  $Cr:\alpha$ - $Al_2O_3$  transparent ceramics with vacuum sintering method. The concentration of  $Cr_2O_3$  in the present study ranged from 0.10 wt% (by weight) to 0.40 wt%. 0.10 wt%  $La_2O_3$  was introduced as a sintering aid. The powders were mixed in absolute ethyl alcohol for 24 h with zirconia balls, and then synthesized by conventional solid-state reaction at 1100 °C for 24 h in air atmosphere. Disks with 25 mm in diameter and 10 mm in thickness were isostatically pressed at 200 MPa. The samples were sintered at 1700 °C for 10 h under vacuum conditions. Finally the specimens were cut and double polished with 1-2 mm in thickness for spectral analysis.

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Microstructures were observed with scanning electron microscope (SEM, JSM-6700F, JEOL, Japan). The fluorescence and excitation spectra were measured with a spectrofluorimeter (FP-6500/6600, JASCO), of which the light source was 150W Xe light and the resolution was 1-2nm. The TL and OSL measurements were carried out on RisФTL/OSL-DA-15 automatic reader (RisΦ National Laboratory, Denmark) from an integrated <sup>90</sup>Sr beta source. The TL tests were performed ranging from 273 K to 550 K at a heating rate of 2 K/s in a vacuum chamber with a beta dose of 1 Gy. The U340 optical filters of thickness in 2.5 mm are used for the light detection. The excitation light is a high intensity blue light emitting diode cluster with wavelength 470 ± 30 nm.

#### 3. Results and discussion

Fig. 1 is Photograph of  $Cr:\alpha-Al_2O_3$  transparent ceramics doped with 0.1 wt%  $Cr_2O_3$  (1.20 mm in thickness). The specimen has high transparency and the letters under the ceramics can be seen clearly.

Fig. 2 shows the SEM microstructure photographs of  $Cr:\alpha-Al_2O_3$  transparent ceramics doped with different content of  $Cr_2O_3$ . It reveals that the polycrystalline  $Cr:\alpha-Al_2O_3$  transparent ceramics almost have no pores and the fractured surface of specimens is intergranular broken section. Grain size remarkably decreases with increasing concentration of  $Cr_2O_3$ . This indicates that  $Cr_2O_3$  can effectively inhibit excessive grain growth of  $Cr:\alpha-Al_2O_3$  transparent ceramics.

The absorption spectra of specimens doped with 0.1 wt%  $Cr_2O_3$  is shown in Fig. 3. Two absorption peaks are apparent in the absorption spectra of specimens which are assigned to the emission of  $Cr^{3+}$  ions. One peak lies at 402 nm due to  ${}^4A_2 \rightarrow {}^4T_1$  transition and the other one at 558 nm due to  ${}^4A_2 \rightarrow {}^4T_2$  transition. The absorption peaks agree fairly well with the absorption spectrum of  $Cr:\alpha$ - $Al_2O_3$  single crystals [17,18].

The fluorescence spectra of Cr:α-Al<sub>2</sub>O<sub>3</sub> transparent ceramics doped with 0.1 wt% Cr<sub>2</sub>O<sub>3</sub> at room temperature is shown in Fig. 4. It shows emission peak at 670 nm and 697 nm after stimulated at 578 nm at room temperature. It is well known that the spectra of Cr:α-Al<sub>2</sub>O<sub>3</sub> single crystal [18] shows the well-known narrow R-line that consists of two closely spaced lines,  $R_1$  and  $R_2$ line, located at 693 nm and 694 nm at room temperature, which are due to emission from Cr3+ ions substituted et al. sites. By and large, emission peak at 697 nm of Cr: $\alpha$ -Al<sub>2</sub>O<sub>3</sub> transparent ceramics nearly equal to that of Cr:α-Al<sub>2</sub>O<sub>3</sub> single crystals. Besides, it shows a broad band emission in the range of 690-705 nm. On the one hand, the crystal structure of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> belongs to a trigonal system and is optically uniaxial and birefringent. From the macroscopic viewpoint, grains of α-Al<sub>2</sub>O<sub>3</sub> ceramics are distributed homogeneously and isotropically, whose emission spectra vary with each other. The broad band emission in the range of 690-705 nm results from the accumulating emission peaks of different grains. On the other hand, various defects located at grain boundary lead to lattice deformation and give rise to the broad band emission. The emission peak at 670 nm in Cr:α-Al<sub>2</sub>O<sub>3</sub> transparent ceramics was never seen in previous studied [17-20]. This may be explained by the introduction of La<sub>2</sub>O<sub>3</sub>, which would change the positions of  $Cr^{3+}$  ions in  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> octahedral structure.

TL curves of  $Cr:\alpha-Al_2O_3$  transparent ceramics with different content of  $Cr_2O_3$  with a dose of 1 Gy is shown in Fig. 5.



Fig. 1. Photograph of  $Cr:\alpha$ -Al<sub>2</sub>O<sub>3</sub> transparent ceramics doped with 0.1 wt%  $Cr_2O_3$  (1.20 mm in thickness).

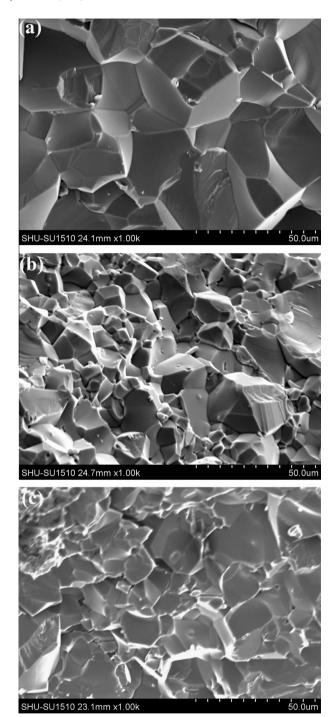


Fig. 2. SEM photographs of  $Cr:\alpha-Al_2O_3$  transparent ceramics doped with different content of  $Cr_2O_3$  (a) 0.1 wt%  $Cr_2O_3$ ; (b) 0.2 wt%  $Cr_2O_3$ ; (c) 0.4 wt%  $Cr_2O_3$ .

Cr: $\alpha$ -Al<sub>2</sub>O<sub>3</sub> transparent ceramics show two different TL glow peaks, a small peak at 397 K and a dominated one at 503 K, which basically agrees well with Cr: $\alpha$ -Al<sub>2</sub>O<sub>3</sub> single crystals [21]. However, the positions of TL peaks shift to higher temperature comparing to Cr: $\alpha$ -Al<sub>2</sub>O<sub>3</sub> single crystals with TL peaks at 110 °C (383 K) and 225 °C (498 K). Besides, Cr: $\alpha$ -Al<sub>2</sub>O<sub>3</sub> single crystals also show peaks at 160 °C (433 K) and 270 °C (543 K). It may be explained by the difference structure and ion concentration between the polycrystalline Cr: $\alpha$ -Al<sub>2</sub>O<sub>3</sub> transparent ceramics and Cr: $\alpha$ -Al<sub>2</sub>O<sub>3</sub> single crystals. This result reinforces the assumption that Cr<sup>3+</sup> ions responsible for the glow peaks. That is to say, TL glow peak positions are impurity dependent. The TL intensity increases with

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