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Luminescence characterisation of alumina substrates using cathodoluminescence microscopy and spectroscopy

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HIGHLIGHTS

- Morphological (SEM) and elemental characterisation (EDS/WDs) of alumina substrates.
- Cathodoluminescence (CL) emission spectroscopy of alumina substrates.
- Close relationship of the CL emission with the SEM and EDs/WDS characteristics.

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ABSTRACT

Polycrystalline alumina (Al_2O_3) substrates, found in many electronic devices and proposed as dosemeters in emergency situations, were invstigated using a scanning electron microscope (SEM) equipped with cathodoluminescence (CL) and elemental analysis probes. The characteristics of the CL spectra, surface morphology, and impurity content of the Al_2O_3 substrates were examined and compared with those of single crystal dosimetry-grade Al_2O_3:C. Whereas the CL spectrum, measured from 250 to 800 nm, for the Al_2O_3:C, contained resolved bands located at ~340 nm and at ~410 nm, the spectrum measured with the Al_2O_3 substrate was significantly broader, extending from ~250 to ~450 nm, and also included a narrow band at 695 nm. While it is likely that the accepted model of recombination at F^+ (~340 nm) and F (~410 nm) in Al_2O_3:C also applies to the substrate, it is suggested that the presence of impurities within the alumina give rise to additional recombination centres. The 695 nm emission has been assigned to a Cr^{3+} ion impurity in previous work on alumina and a band indicated at ~300 nm may be associated with Mg^{2+} or Ca^{2+}, the presence of which was confirmed by elemental mapping. Comparison of the spatial distribution of CL with the surface morphology and elemental composition of the samples indicates that the components of the emission spectrum can be qualitatively correlated with impurity content and morphological features of the samples.

1. Introduction

Aluminium oxide, or alumina, is a ceramic material with high technological interest. Its applications span from the field of substrate manufacturing to the area of radiation protection. Owing to its electrical insulation properties, alumina has been the preferred material for the construction of the substrates of passive electronic components (e.g., resistors and capacitors) incorporated in integrated circuits. A more recent application of these substrates, when embedded in personal electronic equipment devices such as mobile phones, is in the field of accident/retrospective dosimetry and their suitability as emergency dosemeters has been investigated by various authors (Inrig, 2008; Beerten, 2009; Ekendahl and Judas, 2012; Pascu et al., 2013). The potential for the use of alumina substrates in accident dosimetry relies upon the high availability of these components in electronic devices, the linear dose response over the range 0.01–100 Gy, and the possibility to detect doses as low as 20 mGy. The alumina substrates, formed by sintering powdered Al_2O_3 with additives (Topfer, 1971; Minges, 1989; Soni et al., 1995; Altay and Gulgum, 2003), share some of their basic characteristics with dosimetry grade Al_2O_3:C. There are, however, notable differences, in particular the presence of athermal fading (Inrig, 2008; Ekendahl and Judas, 2012) which results in only approximately 50% of the TL/OSL signal remaining after few days following the irradiation. Since the physical form of the substrates

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and the processes associated with the emission of luminescence have previously not been examined in detail, we investigated these aspects using surface probing techniques available with a scanning electron microscope (SEM) with the aim ultimately of contributing to the development of a model for luminescence processes in alumina substrates.

2. Materials and methodology

As part of a wider study, samples of surface-mount resistors with alumina substrates obtained as unused components from different manufacturers and used components extracted from mobile phones have been found to have similar microscopic, spectroscopic and elemental characteristics. The results reported in this paper were obtained with an unused type 1206 resistor (typically 2.1 mm by 0.5 mm thick). Dosimetry-grade Al_2O_3:C chips (Laundauer Stillwater Crystal Growth Division, Stillwater, OK, USA) were also tested for comparison; they are in the form of discs of ca ~5 mm diameter cut from single crystal rods using a diamond-impregnated wire saw.

All tests were carried out using an Hitachi SU-70 FEG SEM. The surface morphology of both untreated and polished samples was investigated using the secondary electron (SE) and backscattered electron (BSE) imaging modes of the SEM and the chemical environment was analysed using energy- and wavelength-dispersive X-ray spectroscopy (EDS and WDS) attached to the SEM system. CL emission spectra were collected using a coupled MonoCL3 (Gatan Ltd, Oxford) scanning monochromator that incorporates a PMT detector (Hamamatsu R374) operated in single photon counting mode and a 1200 lines/mm diffraction grating; the entrance and exit slits of the monochromator were set to 5 mm (30 nm FWHM). All spectra were acquired with an acceleration voltage of 10 kV, a step size of 0.5 nm, and a dwell time of 1 s. The combined measurement of cathodoluminescence (CL) spectral and topographical analysis of the same specimen allowed an examination of the relationship between the stimulated luminescence and the physical form of the substrate matrix.

3. Results and discussion

3.1. Surface morphology

Selected micrographs of the polished and untreated alumina substrate samples are presented in Fig. 1. The images of the same regions show untreated surfaces (a, b) and polished surfaces (c, d) obtained in SE (a, c) and BSE (b, d) measurement modes. The surface of the untreated alumina substrates is heterogeneous, consisting of irregularly shaped grains that are randomly oriented. Grain boundaries and pores can also be identified. Although the grain structure is less obvious in the surface of the polished sample, a random distribution of voids indicated by darker areas is revealed and bright spots can also be observed distributed across the sampled surface. As explained later, the differences in contrast in the BSE image indicate different elemental concentrations. The dosimetry-grade α-Al_2O_3 single crystals, supplied as cut chips, were polished to obtain a flat homogeneous surface. Comparison of the BSE images (not shown) of both the polished and untreated surfaces did not reveal significant compositional variation between the surface and the interior.

3.2. Elemental analysis

The elemental composition of the samples was analysed using a combined EDS-WDS method and employing an electron beam voltage of 15 kV. The semi-quantitative elemental analysis confirmed that the dosimetry-grade Al_2O_3:C crystal was free of major impurities with weight percent concentrations of 57% aluminium and 43% oxygen and that the alumina substrate contained 97% Al_2O_3 (i.e., 53% aluminum and 44% oxygen) and 3% of other oxygen compounds based on the elements Mg, Ca, and Si.

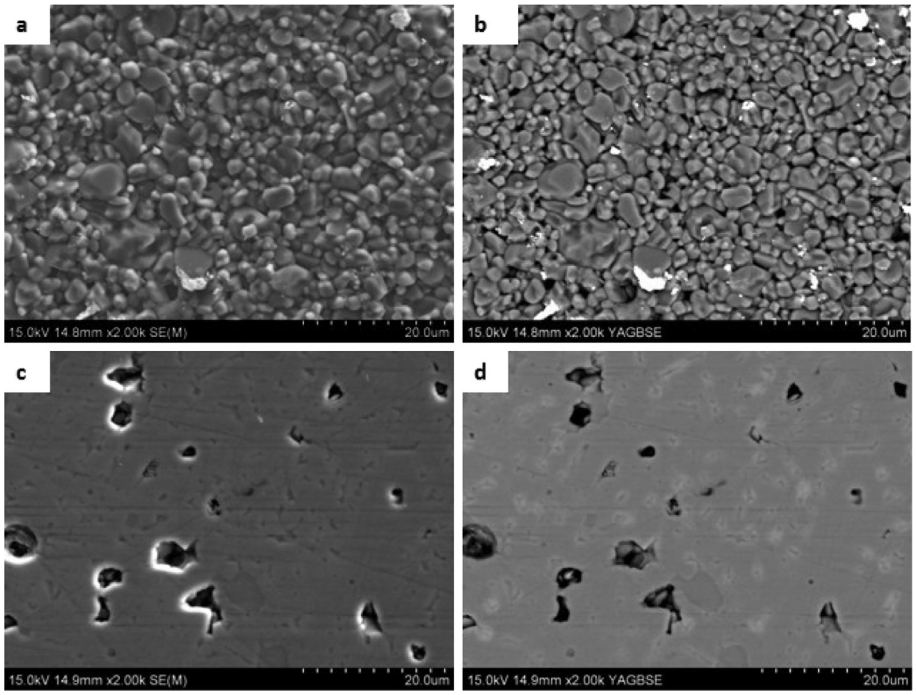

Fig. 1. SEM micrographs of alumina resistor substrates recorded at 2000 magnification. The images of the same regions show untreated surfaces (a, b) and polished surfaces (c, d) obtained in SE (a, c) and BSE (b, d) measurement modes. The images were acquired with an accelerating voltage of 15 kV and electron probe current of 0.5 nA.