

White beam μ -XRF based REE analysis of mineral inclusions in a Kankan diamond

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Introduction

The capability of using the full bending magnet spectrum (white beam) at beamline L allows the high energy XRF based determination of Lanthanide rare earth elements (REE) by their K fluorescence emission lines. This work illustrates an example of spatially resolved REE analysis of a mineral inclusion within a Kankan diamond, originating from Guinea, West Africa.

Inclusions in natural diamonds can provide direct information on the physical and chemical conditions in the deep Earth down at least to the upper part of the lower mantle [1-2]. Only a few sources, e.g. Juina (Brazil) and Kankan (Guinea), provide diamonds from superdeep sublithospheric mantle regions of the deep Earth which originate at depths of several hundreds of km. Determination of the trace-level rare-earth elements composition of the mineral phases in the diamonds can allow the determination of the inclusions' origin (sedimentary, hydrothermal, metasomatic or carbonatitic).

Experimental

The experiments were performed at beamline L. In order to access the K fluorescence lines of the lanthanide rare earth series, the full bending magnet excitation spectrum was used. The low energy contribution of the white beam was reduced by using a 200 μm Al and a 500 μm Cu filter. A beam size of 10 μm on the sample was achieved by using a 10 μm parallel bore hole glass capillary. The fluorescence X-rays were detected with a LN₂ cooled Ge detector (Gresham Gemini 30, 30 mm² crystal area, 3 mm crystal thickness, measured FWHM on Mn K α : 276 eV) coupled with a XIA DXP-XMAP electronics.

On the optically detectable inclusions within the diamond high energy μ -XRF maps were collected with a scanning step-size of 5 μm and measuring times of typically 3-12 s per pixel. On the elemental maps showing the presence of rare earth elements, point measurements with a measuring time of 1000 s were taken.

XRF quantification

The elemental concentrations were determined by using the fundamental parameter quantification method, based on measured elemental yields from a NIST SRM612 (thickness of 100 μm) trace-element glass standard. During the quantification procedure, the beam absorbers, the absorption of both the exciting and fluorescent photons, as well as the polychromatic nature of the beam was taken into account. The error estimates were calculated using a Monte Carlo based approach: the initial parameters for the fundamental parameter method (standard reference material weight fractions, sample densities, XRF line intensities etc.) were varied within their expected statistical deviations and measurement errors that spawned new parameters according to Poisson, uniform or Gaussian distributions. These new parameters were then used to obtain a new set of elemental concentrations. Repeating this procedure several tens of thousands times produces a reliable distribution of calculated elemental concentrations. The standard deviations of the resulting distributions are used to calculate the corresponding confidence intervals.

Results

Elemental distributions for the rare earth elements La, Ce, Nd and Th are given in Figure 1, and a long point measurement taken at a La and Ce rich region is given in Figure 2. The quantified concentrations for this spectrum are summarized in Table 1.

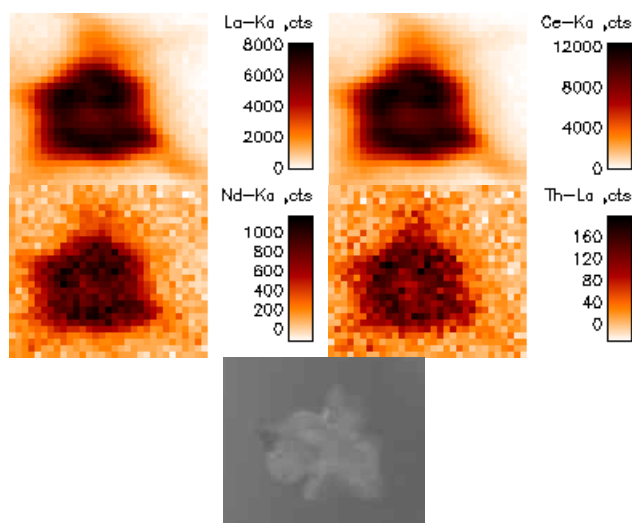


Figure 1: top: elemental μ -XRF maps for the rare earth elements ($27 (5 \mu\text{m}) \times 31 (5 \mu\text{m})$, 15 s); bottom: optical image of the inclusion within a Kankan diamond (KK200, inclusion #4).

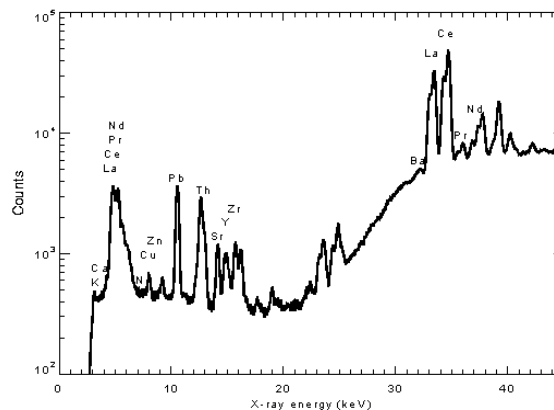


Figure 2: Point measurement on a Ce and La rich area, RT=1000 s.

Table 1: Quantified areal concentrations for the detected rare earth elements..

Element	Areal concentration ($\mu\text{g}/\text{cm}^2$, $\pm 1 \text{ SD}$)
La	143.1 (± 15.9)
Ce	220.0 (± 23.9)
Pr	9.5 (± 1.1)
Nd	24.1 (± 2.7)
Th	10.3 (± 1.3)

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