

OPTICAL CATHODOLUMINESCENCE FOR THE STUDY OF INTERNAL TEXTURES OF ZIRCON

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In recent years the importance of a thorough investigation of the internal textures and (where possible) the distribution of minor elements in zircon crystals used for U-Th-Pb or Pb-Pb dating has become very clear. Cathodoluminescence (CL) represents one of the means for texture investigations. We present here our experience of the use of the Luminoscope, a simple and robust accessory easily mounted on conventional optical microscopes, for this type of study. We examined about 75 zircon concentrates each containing from around 10 to more than 30 grains, about 20 large single crystals, as well as numerous uncovered or polished thin sections and rock slabs. The second author contributed to the study of about 30 of the grain concentrates.

The grain concentrates were usually prepared by magnetic separation of heavy mineral fractions, hand-picked according to shape, colour and extent of fracturing. Heavily metamict zircon grains are usually excluded during the hand-picking process, so we have had no opportunity to study the effects of metamictization on CL patterns, and the possibility that U/Th act as activators for CL.

The rock samples include medium- to high-grade, polymetamorphic gneisses and granulites of Archaean and Proterozoic ages, and a few mainly Neoproterozoic, mostly granitic plutonic rocks. A few of the results have already been published (McReath et al., 2002) or otherwise communicated, and others are presented in detail in other communications to this symposium.

CL is excited when an electron beam is directed onto suitably activated crystals. The light usually originates from the top 2-3 µm layer, and may be observed visually and recorded by conventional or digital cameras, or analyzed by a spectrometer. We use a Luminoscope ELM-3R made by the Nuclide Corporation, which operates stably with electron beams generated by excitation at ≤15 kV and ≤1 mA, producing focused elliptical spots with diameters between a few mm and a few cm. Although rustic, with totally manual operation of potentiometer controls and selector switches, with meter readouts of operating conditions of the power supply, and only simple controls for X-Y adjustment of the sample position in the vacuum chamber, the equipment is easily serviced since the electronic circuits are simple and contain components which have equivalents easily found in Brazil, and the vacuum chamber has very simple mechanics easily maintained by machine shop technician.

Sample grains are mounted in epoxy resin discs. The resin burns under higher power loads, so most CL images

are obtained at 7.5 kV/0.4 mA using a defocused beam with diameter around 1 cm. Under certain conditions (see later), the resin appears to be reflective/refractive, but is sufficiently clear to permit examination of the grains in transmitted light.

Microscopes used for visual/photographic observation include a zoom stereomicroscope for large crystals up to about 1 cm, and a binocular research or a simple monocular microscope for other imaging or spectrometric work. For the latter, best results are obtained with large aperture objective lenses and high transmission "straight through" optical systems with the minimum of lenses or prisms between the objective and the eyepiece. We find the Olympus UMPlanFl 5x objective with an effective aperture of 0.15 to be adequate, but the Gamma Scientific 700-10 -4A 2.9x objective with adjustable effective aperture up to 0.36 is better when the emitted CL is weak. The necessary combinations of effective aperture, working distance between objective and sample, and the geometry of the Luminoscope vacuum chamber severely restrict the choice of objectives. So far we have found only one 25x objective which is satisfactory for imaging purposes, but not for spectrometry since its effective aperture is very low.

The spectrometer is a dual grating ARC SpectraPro 150, connected to the microscope using a special ocular with a collecting fibre which transmits the light to the spectrometer via glass fibre. The sampled area is between about 50 µm and 150 µm, depending on the ocular magnification and the fibre diameter. The digital camera is a ~0.3 megapixel Media Cybernetics Pro-Series 3 chip color camera with appropriate capture kit which allows realtime visualization of the field-of-view, which is about 0.92x0.68 mm with a 5x objective and a 3.3x camera eyepiece, while that for the 2.9x objective with a 10x eyepiece is 1.4x1.0 mm. With an adequately prepared resin mount, these fields-of-view allow the simultaneous observation of number of zircon grains. Maximizing the optical and digital magnifications up to the limit in which pixels appear allows the enlargement of an image of a 200 µm long crystal to around 20 cm.

A lack of CL in zircon is often observed, at least in parts of, if not in the whole crystal. Pale or intense blue, deep to medium violet, yellow, yellow-orange and green CL colours have all been reported (Marshall, 1988) although the identification of the activator elements is sometimes far from unanimous. In contrast, images obtained with most CL attachments for microprobes or SEM are only false color, usually rendered in black and

white. Zoning is also a common feature. Repeated fine- to medium-scale concentric zoning around a euhedral core is common in zircon crystals in igneous rocks, and is frequently preserved with slight modification in the form of zone broadening after medium- to high-grade metamorphism. The latter feature has also been recorded by Connolly (2000). Most of the samples with these features which have also been analysed for the U/Pb system are slightly to strongly discordant. The repeated CL zoning may mimic color zoning in transmitted light. Variations in the widths of dark and light bands are common, ranging from a type with broad dark bands and very thin bright bands to another with broad bright and dark bands have been observed. Repetitions of zoning patterns vary from abundant to rare. In cases where few repetitions are present, the zoning may appear to be restricted to parallel bands, though this pattern could also be a consequence of grain fragmentation. All these features have been described by Hoskin (2000) in his systematic study of the evolution of zoning patterns in zircon from a zoned pluton. At present, we have had no opportunity to perform a similar study. Sector zoning including the hourglass type is also common in zircon from kimberlites, and hourglass zoning is sometimes seen in other igneous and in metamorphic rocks. In a few of these cases, the colour zoning in transmitted light repeats the CL pattern.

Various special situations affect the validity of zircon geochronology: (i) the presence of inherited cores older than the crystallization age of an igneous rock; (ii) the presence of overgrowths younger than crystallization or metamorphic ages; and (iii) redistribution of material by leaching along fractures or other mobilization processes. Exactly what constitutes an inherited core is not always obvious, but we believe that a few unequivocal cases with different characteristics have been observed. Examples of elliptical nuclei without CL have been seen for which the long axes do not coincide with the axes of the repeatedly zoned external part. Cracked nuclei with brighter CL and zoning patterns different from those in the overgrowths are sometimes observed. In transmitted light these nuclei are darker and apparently more metamict than the zoned part.

The identification of overgrowths is made difficult when the preparation of the grain mount is imperfect. Ideally, the grains should be ground down to "waist level" so that the contacts between grains and resin are vertical. Where "waist level" is not reached the contacts are inclined and produce shadows over the unexposed portions of the crystals which are usually seen in transmitted light images, while the edges of the resin may appear as thin, bright and continuous CL lines symmetrical with the crystal outlines. This might be confused with a thin overgrowth. For this reason, each grain mount should contain only fractions of similar grain

size, and the preparation should include grinding and polishing in stages, with checks on the adequacy of the mount after each grinding stage until a satisfactory finish is achieved. What we believe to be overgrowths frequently do not completely surround the rest of the grain, and may be brighter or darker than the edge of the main mass of the grain. The outline of the main overgrown grain may or may not be rounded at the apexes. Another consequence of not attaining an adequate depth of cutting is that the working face may be dominated by a single exposed igneous zone and due to the shallow depth of origin the CL image will appear to be rather uniform.

Fractures are often accompanied by the interruption of repeated CL zoning. They may be darker or lighter than the underlying CL pattern. Patches of darker or lighter material may sprout from the fractures. These features may sometimes be seen in the transmitted light images.

Ideally, the CL studies should be complemented by the obtention of back-scattered electron images and, where possible, by secondary X-ray maps of the grains for elements such as Hf, U, Th etc. In the few cases that we have been able to study in detail, it is clear that zoning of other elements does not necessarily follow the zoning of CL activator elements. Fractures may be accompanied by local loss or gain of one or all of the other analysed elements, and the anomalous patches representing either deficiencies or concentrations of the element are also present. It has not yet been possible to check whether these features are also accompanied by changes in U/Pb ages, or in the degree of concordance.

Although monocrystal Pb-Pb evaporation or U-Pb geochronology using grains selected by optical CL will obviously not substitute SHRIMP or other point analysis methods, the combination offers a useful alternative for dating of zircon grains which do not present registers of complex evolution.

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