Experimental methodology to study the luminescence emission of a pegmatitic feldspar for dosing assessment purposes

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ABSTRACT

The luminescence of quartz and feldspars is usually employed in the field of geological and archaeological dating. The experimental methodology to obtain the utmost information of the samples will depend on the features of each specimen. This paper focuses on the different techniques to be used for the study of pegmatitic K- feldspar that could be suitable for age determination. Previous analysis on the structural characteristics and the chemical content of impurities in the sample, provide information about the wavebands that is of great interest to select the adequate experimental conditions.

Keywords: *Pegmatitic feldspar, Luminescence, Emission bands.*

INTRODUCTION

The luminescence emission of quartz and alkali aluminosilicates is usually employed in the field of geological and archaeological dating. The experimental methodology used for such purpose is based on the assumption that the emission of the mineral is caused by the absorbed radiation dose, from the environmental radioactivity (mainly due to ^{238}U, ^{232}Th and ^{40}K), from the last rigorous heating. In the case of geological dating it corresponds to the time of formation and for archaeological dating the time since last firing. The luminescence process is produced when radiation (optical light, UV light, electron beams, alpha, beta, gamma, X-rays…) strikes the sample. Some of the radiation energy is absorbed and, subsequently, re-emitted (usually) at longer wavelengths. Such emission only depends on the nature of the sample. The specific information extracted from the sample is important to define not only the experimental methodology, but also to a rough idea about the potential wavebands and relative intensity of the luminescence signal. This paper focuses on the study of a pegmatitic K-feldspar based on (i) the structural analysis by polarizing microscopy and X-ray diffraction (XRD), (ii) chemical analysis by electron probe microanalysis (EPMA) and X-ray fluorescence (XRF) and (iii) the radio- and thermoluminescence (RL and TL) spectral emission.

EXPERIMENTAL

The sample of perthite microcline, composed of circa 15 % of Na-feldspar and a K-matrix of $Or_{93}Ab_7$, was collected from Corrego Rapa pegmatite (Minas Gerais, Brazil). The XRD pattern (powder method) was carried out using a Siemens D-5000 with Cu K_α radiation at 40 kV and 30 nA. The bulk structural state is that of intermediate microcline (triclinicity $\Delta = 0.84$) but close to that of low microcline. XRF was done in a PHILIPS PW-1404 with an Sc-Mo tube with analyser crystals of Ge, LIF220, LIF200, PE and TLAP and Super-Q manager from Panalytical-Spain as analytical software (Table 1).

The sample was bound together with a polymer and later softly polished offering a flat surface to the EPMA beam. The crystal-chemical characteristics of the feldspar and Na-mappings were determined on data series of electron microprobe analyses (Jeol Superprobe JXA-8900M), bulk and channel-selected (TAP, PETJ, LIF, PETH) X-ray spectra search and by identification routines.

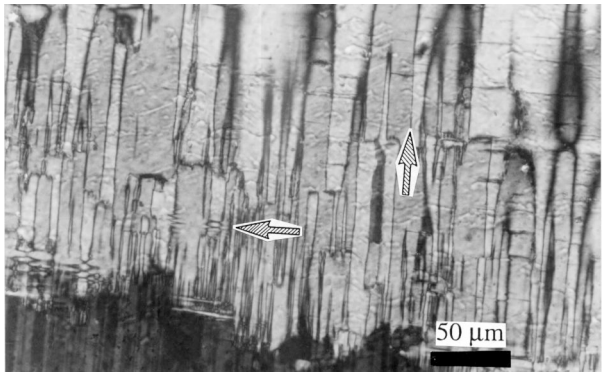

FIGURE 1. Micrograph optical image of the K-feldspar.

TABLE 1. XRF analysis of the sample.

SiO_2	Al_2O_3	Fe_2O_3(t)	CaO	Na_2O	K_2O	P_2O_5	L.I.
66.00	18.20	0.05	0.03	2.34	13.28	0.03	0.2

Rb	Ba	Sr	La	Ce	Y	Mn	Co	Ni	Cr
438	225	38	1	5	2	8	4	4	10

The spot diameter of the probe was circa 5 µm and the operating conditions 15 kV and 20 nA. Spectra were performed on cleaved chips of 3x3x2 mm^3 (~5 mg) of the sample mounted with silicone oil onto aluminium disc using the spectrometer of Sussex University. Signals were recorded over the 200-800 nm wavelength range, with a resolution of 5 nm for 100 point spectra, and 3 nm for 200 point spectra. All signals were corrected for the spectral response of the system. The RL was obtained during excitation of the samples with 50Gy X-irradiated employing a Phillips MG MCN 101 X-ray tube with a current of 15 mA and a voltage of 25 kV delivering a dose rate of 10 $Gy \cdot min^{-1}$ to the sample. TL measurements of mineral coatings were made in the Sussex (UK) TL spectrometer. High sensitivity results from the use of wavelength multiplied detection via a pair of spectrometers, with gratings blazed for the UV-blue (200nm-450nm) and blue-green-red (400nm-800nm) parts of the spectrum, and a pair of position-sensitive photomultiplier tubes. Signals were recorded over the wavelength range 200nm to 800nm, with a resolution of 5nm for 100 channel spectra, and 3nm for 200 channel spectra.

RESULTS AND DISCUSSION

Fig. 1 (previous page) exhibits an optical micrographs of the perthite microcline sample, parallel to (001) using transmitted light, crossed polarizer lens and a deviation of some degrees away from the parallel position. It shows the twinned microcline matrix and a coarse Na-feldspar vein in the lower part with polysynthetic twinning. The twin boundaries of microcline appear as dark lines drawing the domain shape, that are related by the Albite-twin law, being almost equally transparent. The boundaries are generally sharp (arrow 1) but can be "serrated" (arrow 2) by late lattice relaxation and/or twinning substitution. Additionally, a blunting effect on the boundaries produce diffuse bands in which a gradual decrease of the contrast occurs, probably related to submicroscopic super-fine Albite twinning. Irregular twinning at the Na-K interface is absent indicating limited interaction of this microcline with water rich fluids at low temperatures.

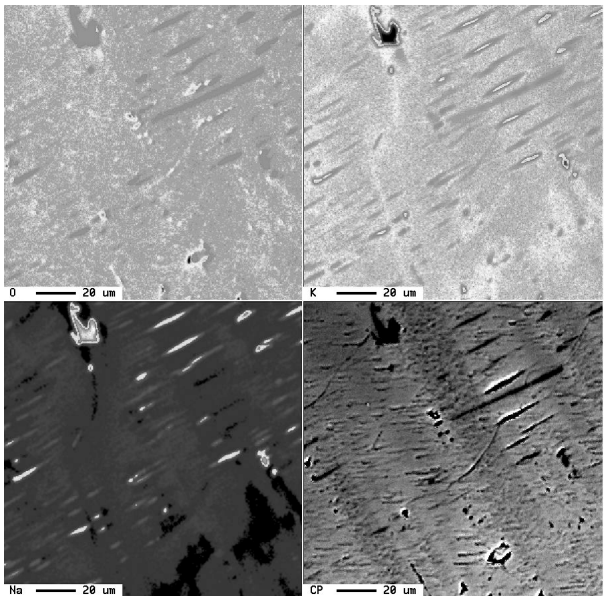

FIGURE 2. Mapping of the sample obtained by EPMA

Fig. 2(a), (b) and (c) are X-ray images collected for O, K and Na atoms respectively from EPMA, in a microcline region (Fig. 2d) similar to that shown in Fig. 1, were obtained by back-scattered electrons, in which Albite twins have different contrast. Fine Na-feldspar domains in a deformational microtexture can be observed indicating that low temperature hydrothermal water rich fluids did not destroy completely the microstructures formed during the subsolidus stage. This information is important because altered K-rich feldspars, in which some water molecules are locally dissolved, behave very different when irradiated in comparison to pristine crystal volumes (Hashimoto et al., 2003).

The luminescence emission (natural and induced TL and RL) spectra are displayed in Fig 3. Four main bands peaked at the UV, blue, green and red region can be appreciated. The higher energy band should correspond to defect-sites associated with the presence of the sodium atoms in the potassium aluminosilicate lattice. The blue emission at 440 nm can be related to Al-O-Al bridges in the lattice, and it is linked to the spatial distribution of elastic strain fields (Sánchez-Munoz et al. 2006). The green band at 550 nm is quite common in all strain-free alkali K-aluminosilicates; being attributed to Mn^{2+} substitutions in calcium sites in the lattice; and the red emission should be attributed to Si (tetrahedrally coordinated) or Al sites can be substituted for Fe^{3+}, acting as recombination sites either for holes or electrons.

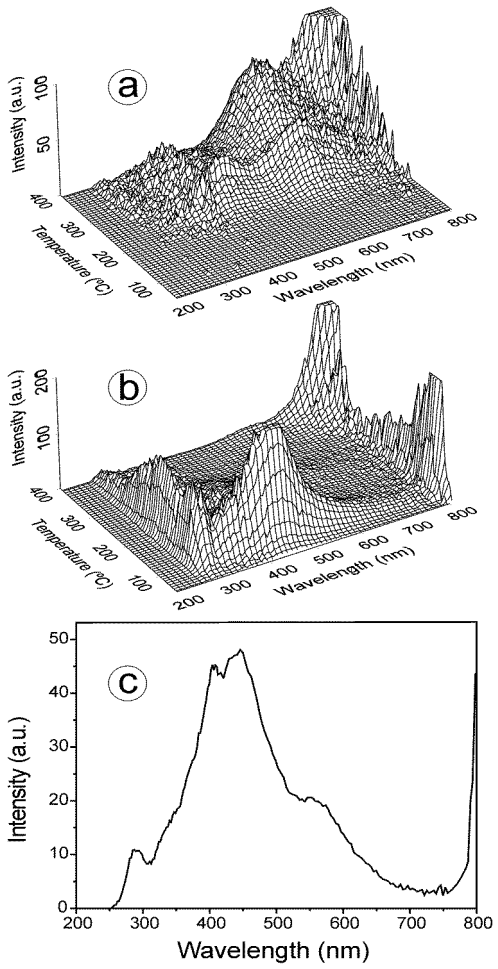

FIGURE 3. 3D TL emission spectra of (a) 'as received' sample (natural TL), (b) after 50 Gy X-ray dose and (c) RL spectrum.

CONCLUSIONS

The chemical analysis of the samples would help in the selection of the appropriate filter to be used in spectral measurements. The similar emission wavebands for the K-rich feldspar indicate that the same recombination centers are active independently of the applied analytical technique: TL or RL.

REFERENCES CITED

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