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# The red luminescence emission of feldspar and its wavelength dependence on K, Na, Ca – composition

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## **Summary**

Feldspar specimens covering the whole Or-Ab-An ternary have been investigated by cathodoluminescence (CL), photoluminescence (PL), radioluminescence (RL) and radiophosphorescence (RP) spectrometry. A red luminescence emission, which is commonly explained by Fe<sup>3+</sup> lattice defects, is a characteristic feature of all the spectra. Different shifts of the peak-wavelength between ~680-750 nm (1.82-1.65 eV) were observed with varying feldspar composition. Despite the dependence of the peak position on the Ca/Na ratio, initially described for CL in the 1970s, there is also a shift induced by changing Na-K composition. The observed effects can be explained by known relations that the peak position of the red luminescence emission in feldspars can be affected both by the structural state of the feldspar and the site occupancy of the trivalent iron. In the case of alkali feldspars another factor may influence the peak-shift. The incorporation of the larger potassium ion causes non-linear variations of the cell dimensions and therefore Fe-O bond distance. The behaviour of the red peak-shift dependent on the feldspar composition is not equal for all types of luminescence investigated. This is most likely caused by the different luminescence excitation mechanism.

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## Introduction

Feldspars show luminescence in different emission wavebands from the UV to the IR. Their appearance and intensity depends not only on the type of feldspar, but also on the stimulation used, i.e. heat (thermoluminescence TL; radiophosphorescence RP, if not induced by tunneling recombination), electron beam interaction (cathodoluminescence, CL), photons (photoluminescence, PL; optically stimulated luminescence, OSL), ionising radiation (radioluminescence, RL) (e.g. Krbetschek et al., 1997). A red glow of feldspar in CL was initially described in the 1960s (Smith and Stenstrom, 1965) and is explained by Fe<sup>3+</sup> lattice defects (Geake et al., 1973; Telfer and Walker, 1975, 1978; White et al., 1986). The emission peak is located between  $\sim$ 680–740 nm (1.82–1.67 eV) depending on feldspar chemical composition. This was initially found during CL measurements to be related to the Na/Ca ratio (i.e. albite/anorthite content) in plagioclases (Sippel and Spencer, 1970; Geake et al., 1973; Telfer and Walker, 1975; Mora and Ramseyer, 1992). Since the dependence of the peak-wavelength of red RP on the potassium concentration of alkali feldspars was firstly measured (Dütsch, 1995) this phenomenon was further investigated (Dütsch and Krbetschek, 1997) and also observed using other types of excitation (RL – Trautmann, 1999; CL – Götze et al., 2000). The present paper summarises the results of investigations carried out using a liquid nitrogen-cooled CCD camera based TL/OSL/RL/PL or CL spectrometers and feldspar specimen characterised by X-ray diffraction or chemical analyses. The PL of single feldspar grains (200–225 μm) has also been investigated. An Ar<sup>+</sup>laser coupled Raman spectrometer and Scanning Electron Microscopy - Electron Dispersive X-ray Analysis (SEM-EDXA) was used for this study.

# **Experimental and results**

# Feldspar samples

A collection of feldspars from igneous and metamorphic rocks and sediments as well as plagioclase grains from lunar soil was used for the different spectral luminescence measurements. Feldspar extraction in the grain size range of  $100-225\,\mu m$  (after crushing and/or sieving) was carried out by heavy liquid density separation. X-ray powder diffraction analyses (24 samples from igneous and metamorphic rocks) and atomic absorption analyses (15 detrital feldspars from sediments) were applied to determine the structural state and/or chemical composition of these samples. The rock-samples were also prepared for light and CL microscopy investigation. Polished thin sections were also applied in PL measurements on feldspar sediment grains. Further analytical detail is given below.

# Spectral luminescence measurements

Three different types of luminescence spectrometers were used, described below in the respective section. The spectra investigated were not corrected for instrument response. Energy shifts of the peak position compared to corrected values are estimated to be small. In the case of photoluminescence measurements the response function of the Raman-spectrometer was determined by measurements using a calibrated tungsten band lamp. The halfwidth (b) of the nearly Gaussian shaped red peak is about 0.24 eV, corresponding to 100 nm for a maximum  $(\omega \cdots \text{peak energy})$  observed at  $\omega_{\text{obs}} = 1.722 \,\text{eV}$  (720 nm). In the energy range  $\omega_{\rm obs} - b/2 \cdots \omega_{\rm obs} + b/2$  a response function that varies linearly can be assumed with changes less than 10%. Therefore, the observed maximum  $\omega_{\rm obs}$  is shifted compared to the correct maximum value  $\omega_0$ . However, the shift is small in comparison with the halfwidth b of the band. In this case the shift can be determined approximately with  $\omega_{\rm obs} - \omega_0 = (b \, m)/(8 \, ln(2))$ , where m is the change of the response function in the energy range  $\omega_{\rm obs} - b/2 \cdots \omega_{\rm obs} + b/2$  (in % divided by 100). Energy shifts of about 0.3% ( $\sim 2$  nm) are obtained. The response function of the luminescence spectrometer used for the RP, RL and CL measurements is also known (Rieser, 1999; Habermann, 1997). The variations between 650 and 780 nm are within  $\sim \pm 5\%$  of the mean sensitivity in this range. Thus the same energy shifts of the red peak, having a halfwidth of about 100 nm in all types of luminescence spectra measured, can be assumed.

There is an individual wavelength calibration of each instrument that can lead to differences in the peak wavelength reported. However, these should also be small, compared with errors caused by sample inhomogeneity and the particular specimen used, i.e. individual feldspar grains, rock slices and thin sections from sediment grains. For each of these, variation relating to the chemical composition determined at a subsample of grains or an individual measurement spot has to be taken into account. Also the different luminescence methods used analyze areas of different size, i.e. spots of  $5\,\mu m$ ,  $30\,\mu m$ ,  $4\,m m$  or  $10\,m m$  diameter in PL, CL, RL and RP measurements, respectively. Because wavelength units are more common in the geological literature, the results are presented in that format. However, fit procedures for determination of peak positions are based on energy units.

## Radiophosphorescence (RP)

RP measurements have been carried out on feldspar specimens from rocks and alkali feldspar fractions from sediments using a LN-cooled CCD camera based luminescence spectrometer system (TL/OSL/RL/PL) with a detection range 200-800 nm (6.20-1.55 eV) (Rieser et al., 1994). The samples (grain size 100-200 μm) were irradiated with 2200 Gy  $^{60}$ Co  $\gamma$ -rays (igneous and metamorphic feldspars) or 50 Gy 90 Sr/90 Y β-radiation (detrital feldspars). After a delay of 1.5–4 hr ( $\gamma$ -irradiated samples) or 1 min ( $\beta$ -irradiated samples) phosphorescence spectra were taken from 8 mg aliquotes on 10 mm diameter sample discs using spectrometer integration times of 5-30 min. The relatively long delay between  $\gamma$ -irradiation and RP measurements was caused by the time needed to transport the sample. The RP decay of the  $\gamma$ -irradiated igneous and metamorphic feldspar specimens has been further investigated over 2 weeks after the initial measurements. All emissions from the UV to the IR have nearly equal signal decay functions and no spectral peak-shifts with time were observed (Rieser, 1999). This indicates that red tunneling afterglow (e.g. Visocekas, 1985) should not significantly influence the measured spectra of these samples.

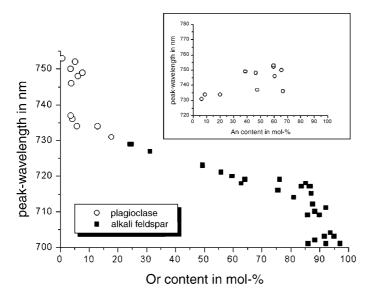


Fig. 1. The shift of the red peak observed in spectral radiophosphorescence (RP) measurements

Figure 1 reveals that the peak-wavelength of the red RP is dependent on potassium concentration. No difference could be observed between the various structural states of potassium feldspar. Thus, the monoclinic feldspar varieties (orthoclase, sanidine) show the same behaviour as the triclinic ones (microcline, anorthoclase). Within the plagioclase series (albite to anorthite) the peak shifts towards longer wavelength with increasing An-content.

## Cathodoluminescence (CL)

CL measurements were carried out on carbon-coated polished thin sections of igneous and metamorphic rocks using a "hot cathode" CL microscope with an acceleration voltage of 14 kV and a current density of  $\sim 10 \,\mu\text{A}\,\text{mm}^{-2}$ . A triple grating spectrograph attached to a LN-cooled CCD camera was used as detection unit, which is linked by a silica-glass fiber optics to the microscope (Neuser et al., 1995). CL spectra were measured between 300–800 nm (4.13–1.55 eV) using standardised conditions of 30 µm spot width and 10 s integration time. Figure 2 shows the results modified after Götze et al. (2000) with additional measurements on lunar samples. Two effects have been observed. At first, the wavelength of the red peak shifts within the plagioclase group depending on the anorthite content as already described in former CL investigations (see section 1.). In contrast to the RP measurements, where plagioclases tend to higher peak-wavelength with increasing anorthite content, the red CL peak shifts from about 740 nm (1.67 eV) to 685 nm  $(1.81 \,\mathrm{eV})$  in the range  $\mathrm{An}_5$  to  $\mathrm{An}_{95}$ . It can be shown that extraterrestrial plagioclases from lunar soil also plot within this correlation. Second, a decreasing peak wavelength with increasing potassium content was detected in alkali feldspars as was observed in RP (Fig. 1).

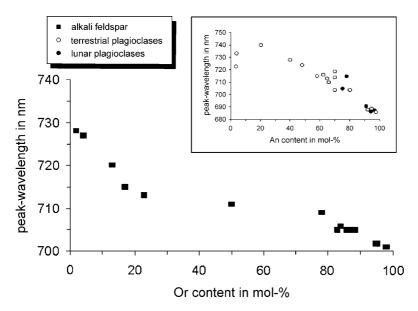


Fig. 2. The shift of the red peak observed in spectral cathodoluminescence (CL) measurements

### Radioluminescence (RL)

Radioluminescence (RL) is stimulated when the samples are excited with ionizing radiation from a radioactive source. For the measurement results presented, a  $^{137}\mathrm{Cs}$  irradiator (3.7 MBq;  $\beta^-$ , conversion electrons) was used for RL excitation. The sample (4 mg, 100–200 µm grains) is placed in a 4 mm diameter sample holder and separated from the radioactive source by a thin (0.02 mm) Al-foil (*Trautmann* et al., 1998). RL light was detected by the same luminescence spectrometer as described above. The spectra were taken with CCD integration times of 300 s. The results (Fig. 3) show a dependence of the peak-wavelength in the red luminescence emission on the potassium concentration of the feldspar specimens. Although the number of measurements is reduced by a lack of sample material caused by former investigations on the feldspar collection shown in the RP and CL results (Figs. 1, 2), a peak-shift is observable. Although this shift is not as regularly shaped as seen for e.g. RP, it behaves similarly. Increasing anorthite content results in longer emission wavelength. This is in contrast to CL results, where Ca-rich plagioclases tend to shorter emission-wavelength (Fig. 2).

# Photoluminescence (PL)

PL investigations were carried out using polished thin sections of feldspar grains (200–225  $\mu m$ ) extracted from Quaternary sediments. Feldspar was separated from quartz by a flotation procedure. The feldspar extract was subsequently divided into three density ranges by heavy liquid separation: 2.53–2.58 g cm $^{-3}$  (potassium feldspar), 2.58–2.65 g cm $^{-3}$  (Na-rich plagioclases) and  $>\!2.65$  g cm $^{-3}$  (Na-Ca plagioclases). From each of these fractions a separate polished thin section has been made.

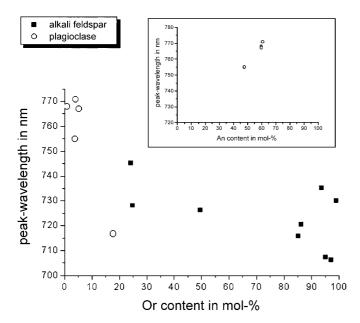


Fig. 3. The shift of the red peak observed in spectral radioluminescence (RL) measurements

The PL spectra were measured with a Jobin Yvon T64000 triple-grating spectrometer equipped with a microscope and a LN-cooled charge coupled device (CCD) detector. The spectra of the samples mounted under the microscope were excited with the 514.5 nm line of an Ar<sup>+</sup>-laser. In order to avoid heating effects of the samples, the laser power was reduced to <4 mW. For analysing the luminescence light, a super notch filter in combination with the single monochromator option of the spectrometer was used. The spectra were measured in the range

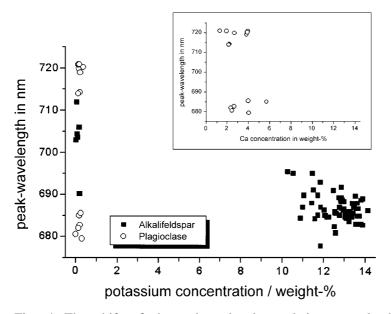


Fig. 4. The shift of the red peak observed in spectral photoluminescence (PL) measurements

 $600-850\,\mathrm{nm}$  ( $2.06-1.46\,\mathrm{eV}$ ). The diameter of the laser focus on the sample was about  $5\,\mu\mathrm{m}$ . The PL measurement spots were marked. After carbon coating of the thin sections, the K, Na, Ca, Al and Si concentrations have been determined by Scanning Electron Microscopy using a Jeol JSM 6400 instrument with Noran energy dispersive X-ray analysis system.

The results of the measurements are shown in Fig. 4. The spot width of the PL (5 µm) and SEM (20 µm) measurements are different and there are also possible errors since it is not possible always to excite exactly the same location on the grain. Unfortunately, among the feldspar samples analysed there were no grains with intermediate alkali feldspar composition. All potassium feldspars show a PL emission in the range of about 680–690 nm (1.82–1.80 eV). There is possibly a trend towards higher wavelength for lower K-concentration. Except for one measurement spot, the peak-wavelength of red PL is between about 705–710 nm (1.76–1.74 eV) for sodium feldspar (<1% Ca). Plagioclases >1% Ca are clearly divided into two groups: 680–685 nm (1.82–1.81 eV) and 715–720 nm (1.73–1.72 eV). Within the investigated range from about 1.3–9% Ca (An<sub>9–36</sub>) there is no detectable dependence on the calcium concentration.

### **Discussion**

The relationships between the chemical composition of feldspars and the peak position of the red luminescence emission observed under different excitations are summarised in Fig. 5. There are three main effects, which occur depending on the type of luminescence (excitation/stimulation mechanism):

- 1) Alkali feldspars show a non-linear shift of the red peak from about  $690 \, \text{nm}$  (Or<sub>0</sub>) to  $740 \, \text{nm}$  (Or<sub>100</sub>) (1.80–1.67 eV) depending on the Or content. This was found for all luminescence types investigated, although the PL measurements do not span the range of intermediate alkali feldspar composition.
- 2) Within the plagioclase group the wavelength of the red cathodoluminescence emission shifts from about  $740 \,\mathrm{nm}$  (An<sub>5</sub>) to  $690 \,\mathrm{nm}$  (An<sub>100</sub>) (1.67–1.80 eV). This trend is almost linear in the range between An<sub>20</sub> and An<sub>100</sub>.
- 3) In RP, RL and PL the peak shift dependent on the An-content (Ca concentration) is different from that observed in CL. The position of the red peak in RP and RL shifts to higher wavelengths with increasing An content (up to 770 nm, 1.61 eV), higher than reached at lowest Or content (~740 nm, 1.67 eV). In PL, plagioclases can be subdivided into two groups based on the wavelength position of the red emission peak. Group one represents plagioclases with low wavelengths of the red emission, comparable to the peak position in K-feldspar. Another group of plagioclases is characterised by high wavelengths comparable to those measured with RP and RL in Ca-rich plagioclases. This effect is independent of the An content, at least in the range investigated between An<sub>9</sub> and An<sub>36</sub>.

The red luminescence emission in feldspars is due to the  ${}^{4}\text{T1} \rightarrow {}^{6}\text{Al}$  transition of Fe<sup>3+</sup> which occupies Al<sup>3+</sup> tetrahedral sites in the lattice (*Geake* et al., 1973; *White* et al., 1986). Fe<sup>3+</sup> substitutes for Al<sup>3+</sup> in the T1 position in ordered alkali feldspars (e.g. low albite) and in the T1 and T2 positions, as well as in tetrahedra of

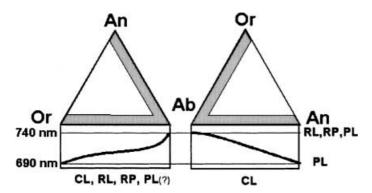


Fig. 5. Schematic summary of the red feldspar luminescence peak-wavelength shift observed in different types of luminescence, dependent on their chemical composition. Within the alkali feldspar group the trend seems equal for the different types of luminescence investigated. The plagioclase series shows a nearly linear trend just in CL measurements. In RL and RP measurements Ca-rich plagioclases emit at high wavelength, whereas in PL emissions in the high and low part of the peak-shift region were observed

type TO<sub>3</sub>OH, in disordered ones (*Petrov* et al., 1989). In plagioclase three lattice positions of Fe<sup>3+</sup> are still under discussion (*Marfunin* et al., 1967; *Boroznovskaya* et al., 1982; *Petrov*, 1994). The peak position of the red luminescence emission in feldspars can be affected both by the structural state of the feldspar and the site occupancy of the trivalent iron (*Telfer* and *Walker*, 1978; *Petrov* et al., 1989).

Finch and Klein (1999) concluded from decay profiles of the red emission in alkali feldspars that the luminescence comprises two components of different half-lives, i.e. that the red band is a combination of superposed but distinct emissions. Correlations between the presence of Fe<sup>3+</sup> lines in the EPR spectra and the position of the CL emission indicated that there exists a relationship between the state of Fe<sup>3+</sup> tetrahedral order. CL emission activated by Fe<sup>3+</sup> on the T1 site was detected in the red region, whereas the emission from the T2 site lies in the IR (Finch and Klein, 1999). Accordingly, red CL derives from Fe<sup>3+</sup> activation in fully ordered alkali feldspars, whereas in disordered or partly ordered feldspars it appears in the IR. Recent results of RL, CL, and ionoluminescence (IL) measurements confirmed the existence of two dominant emissions deriving from Fe<sup>3+</sup>-activated luminescence from low albite and maximum microcline components in alkali feldspar (*Brooks* et al., 2002).

Considering the existence of a solvus in the alkali feldspar system between  $Or_5$  and  $Or_{90}$  at low temperature, the present data in this compositional range (Figs. 1–4) would more indicate the mixture of the luminescence of different amounts of  $Ab_{10}Or_{90}$  and  $Ab_{95}Or_5$  than the existence of single phases. This conclusion would also explain the inflexion at  $\sim Or_{85}$  which is close to the edge of the solvus in the alkali feldspar system. Unfortunately, the mineral composition of the analysed feldspar samples could often only be determined by microchemical analysis, since the samples consist of single feldspar grains. It was not possible to provide firm evidence from XRD that the samples are truly high-temperature monoclinic members of the sanidine-monalbite system.

Another factor that may influence the shift of the red peak in alkali feldspars as a function of chemical composition is the  $K^+-Na^+$  substitution in the crystal lattice (*Schläfer* and *Gliemann*, 1980). The incorporation of the larger potassium ion causes a stretching of the lattice resulting in non-linear variations of the cell dimensions (and therefore Fe–O bond distance). A tentative relationship between the unit cell parameter of feldspar (reflecting Fe–O bond distance) and the emission energy of the red luminescence was also reported by *Brooks* et al. (2002) during temperature dependent CL measurements. This factor may influence the position of the red peak in the compositional ranges between  $Or_{0-5}$  and  $Or_{90-100}$ .

In plagioclases, the sensitivity of the red peak position to anorthite content has early been reported (e.g. *Sippel* and *Spencer*, 1970; *Geake* et al., 1973). There is a large shift of the red luminescence emission, which can probably be related both to structural effects and to the tetrahedral site occupancy of the Fe<sup>3+</sup> ion (*Telfer* and *Walker*, 1978). *Boroznovskaya* et al. (1982) determined various structural sites of Fe<sup>3+</sup> in plagioclase by EPR measurements that may result in different emission maxima of the related X-ray luminescence.

Indications about the existence of more than one emission band in the CL spectra were also drawn from CL measurements of plagioclases with intermediate compositions around  $An_{50}$  (*Götze* et al., 2000). Within this compositional range the existence of incommensurate structures within the plagioclase series results in exsolution phenomena into two phases,  $e_1$  and  $e_2$ , which coexist as lamellar intergrowths of an anorthite-rich and albite-rich component, respectively (*Putnis*, 1992). Variations in the ratio between such phases may cause variations in the intensities of two single bands and result in a shift of the composite red band.

Differences in the intensity and behaviour of the red emission band in dependence on the type of luminescence excitation may be due to different penetration depths of each type of radiation and/or to the efficiency of the generation of different electron trapping centres (e.g. *Townsend* and *Rowlands*, 2000; *Correcher* and *Garcia-Guinea*, 2001).

#### **Conclusions**

The energy/wavelength of the red emission in feldspars is mainly determined by the Fe–O bond distance in the crystal structure (local crystal field). Shifts in the luminescence spectra of feldspar minerals probably relate to several factors that influence this parameter: the structural state of the feldspar crystal, the tetrahedral site occupancy of the Fe<sup>3+</sup> ion, and/or variations in the local crystal field caused by the different ionic radii of cations in the crystal structure due to variations in the chemical composition. Furthermore, overlapping single bands from different structural sites or exsolved components may cause changes of the resulting composite band.

There are differences in the intensity and behaviour of the red emission band in dependence on the type of luminescence. This can be caused by the individual luminescence excitation mechanism. Therefore, the different luminescence types show in general similar luminescence spectra but may vary in specific behaviour during different experimental conditions.

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