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Imaging and measurement of red-infrared stimulated luminescence (R-IRSL) from feldspar samples

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***Abstract**: When the infrared light was illuminated on granites and feldspar slices irradiated with X-rays, luminescence color images (abbreviated to IRSLCI) showed three color patterns, separable into intense yellow, red and faint violet portions. The yellow and faint violet parts were assignable to plagioclase and potassium feldspars and quartz constituent, respectively. The red color parts appeared on both potassium and albite feldspars. From spectrometry of the IRSL, two main emission peaks in the middle wavelength region, consisting of 550 nm (yellow) and 580 nm (orange), were revealed besides intense emission both in wavelengths shorter than 450 nm (violet region) and in wavelengths longer than 600 nm (red region) on every feldspar sample. The red IRSL (R-IRSL) of as-received feldspars was significantly enhanced after annealing treatment in oxidative conditions, rather than in reductive annealing conditions for 3hrs at 900^{o}C. The dose response of R-IRSL offered slower saturation of microcline in comparison with rapid saturation tendency of albite. The results are suggestive of a preferable application of R-IRSL to dating of burnt archaeological materials.*

Introduction

Hütt et al., (1988) have initially reported infrared-stimulated luminescence (IRSL) phenomena for most feldspar samples. When they shone infrared light in the range of 800-900nm on radiation-exposed feldspars, strong IRSL has been observed in visible-light wavelengths. Three kinds of broad luminescence peaks, consisting of strongest 300nm, 390nm in violet regions, and 550nm peaks in yellow-green, have been reported for the IRSL of a low-K albite sample stimulated with light emitting diode (LED) of 880nm emission peak from diode arrays (Clarke and Rendell, 1997). Krbetschek et al. (1997) have reviewed a number of IRSL-emission spectra from feldspars of differing origin and mixtures of various kinds of feldspar.

Recently, the authors have obtained, for the first time, color images of infrared stimulated luminescence (IRSL) for some granite slices, in addition to thermoluminescence color images (TLCI), afterglow color images (AGCI), photo-induced phosphorescence (PIP), color center images (CCI) and 2-dimensional monochromatic OSL-images (Hashimoto et al., 1995, 2002a). In the spectral analysis of IRSL from feldspar constituents, intense red luminescence emission has been shown in wavelengths longer than 600nm, as well as 550nm and 580nm broad peaks.

Since the IRSL from feldspars is very effectively bleached with sunlight at deposition (Aitken, 1998), the IRSL-dating of feldspars is a useful addition to the dating of Quaternary-sediment layers. In the present paper, the imaging technique of red-IRSL (R-IRSL) phenomena was developed for feldspar samples and a granite slice. Subsequently, further quantitative measurements of R-IRSL were carried out after reduction of background events as low as possible by means of the careful selection of filter-combination. R-IRSL enhancement from two kinds of feldspars has been examined by applying thermal annealing treatments in oxidative or reductive environments

Experimental

1. Preparation of samples

Slice and grain samples of granite rock and two kinds of feldspars, were prepared from: 1) An HW-2 granite, mylonite-like sub-facies (granodiorite), originated from Hanawa pluton zone adjacent to Tanakura Shear Zone, Fukushima, Japan, 2) a microcline (potassium feldspar) was of pegmatite origin, Nellore, Andhra Pradesh, India, and 3) an albite feldspar was obtained from Itoigawa, Niigata, Japan. The HW-2 specimen has almost the same origin as sample HW-5 in a previous paper (Hashimoto et al., 2002a).

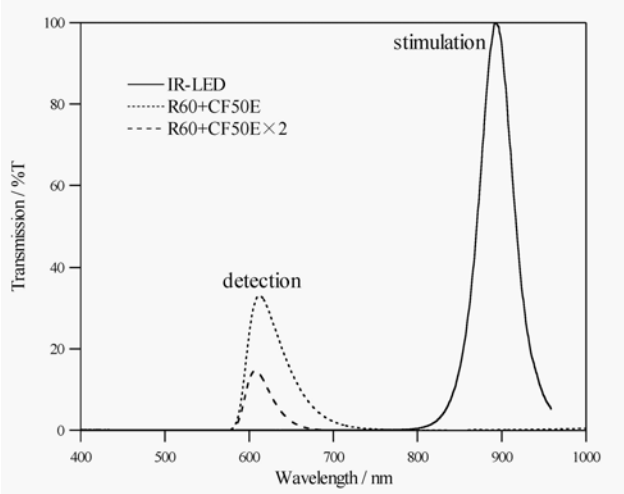

Figure 1.
Spectral response of infrared from light emitting diode (LED) for stimulation and filter assemblies for red-IRSL detection.

After cutting roughly into rectangular planes (approximately 30x20x0.5 mm), each surface was polished with an alumina emulsion solution. Small feldspar disk slices (less than ϕ=5mm) were subjected to IRSL-spectrometry. Every sliced rock sample was followed by X-ray irradiation. The feldspar pieces were also crushed to prepare grain samples (ϕ=150-250μm) for the measurements of R-IRSL decay curves associated with thermal annealing treatments. Thermal annealing treatment was performed in oxidative and reductive conditions for 3 hr at about 900°C. To attain reductive conditions, the inner atmosphere of the quartz capsule containing the grain samples was replaced with nitrogen gas and annealing was achieved in the presence of graphite powder. In the case of oxidative conditions, the sample grains were annealed in atmospheric air.

2. Observation of IRSL color images and spectrometry

All luminescence color images were observed after irradiation from an X-ray generator (Philips, PW 1830), of which the exposure rate was standardized with a ^{137}Cs standard γ-ray source (Pony Co. Ltd., PS-3000 SB Type). Following X-ray irradiation with an absorbed dose of 200-800 Gy (for 5-20 min exposure time), slice samples were "cooled" to remove completely afterglow emission, by letting the samples stand for 1 day or more in a dark box. The IRSL-color imaging (IRSLCI) observation of the slice samples was carried out by means of a photographic assembly, interposing a cold filter (Kenko CF) between camera and sample, as described in Hashimoto et al. (2002a).

An on-line spectrometric system for extremely weak photon-emission was used for the spectrometry of IRSL from pieces of feldspar slice in the similar way to the granite ones (Hashimoto et al., 1997; 2002a). Every scanning interval was 22 msec and 512 channel data (in wavelength) were summed up to 45 cycles to form one spectrum per second. Thus, 100 spectra during the period of 100 sec stimulated with IR-light, could be acquired to the microcomputer memory. All of the spectrum data were plotted in a spectrum for every slice sample.

3. Red IRSL measurement

Sixteen LEDs (IR-LED, Hamamatsu Photonics, L2690-02), with 890 nm emission peak with 50 nm FWHM value, were installed to an LED holder having a hole of 10 mm in diameter. This IR-LED holder was fixed to an automatic system for the TL and OSL measurement equipped with a small X-ray irradiator (Hashimoto et al., 2002b). The applied IR power was measured to be 27 mW/cm^2 at the surface of the grain samples in a silver pan (ϕ=8mm), in which about 5 mg of feldspar grains was placed for every measurement. The optical properties in transmission of the filter combination and stimulation light from the IR-LED are shown in Fig. 1. To keep background events as low as possible, the transmittance of stimulated IR-LED light should be excluded completely from the low-wavelength tail of the 890nm-emission peak. A photomultiplier tube (Hamamatsu Photonics, R-649S) installed in a cooling box at -20°C is sensitive to long wavelengths owing to a multialkali surface. Since this photomultiplier tube is sensitive even to infrared light, two cold filters (Asahi-techno Glass, CF-50E) were used. In these conditions, the mean background counting rate under the illumination of 16 IR-LEDs amounted to 580 counts per 0.1sec data acquisition period.

All R-IRSL measurements were carried out at 125°C after preheating for 3min at 220°C after the artificial irradiation. The dose-response curves were obtained using a single aliquot regenerative (SAR) method (Aitken, 1998) with correction for sensitivity change using a test dose of 10.5Gy.

Results and discussion

1) IRSL color images

Infrared-stimulated luminescence color images (IRSLCI) of the X-ray-irradiated granite and microcline slices are presented in Fig. 2.

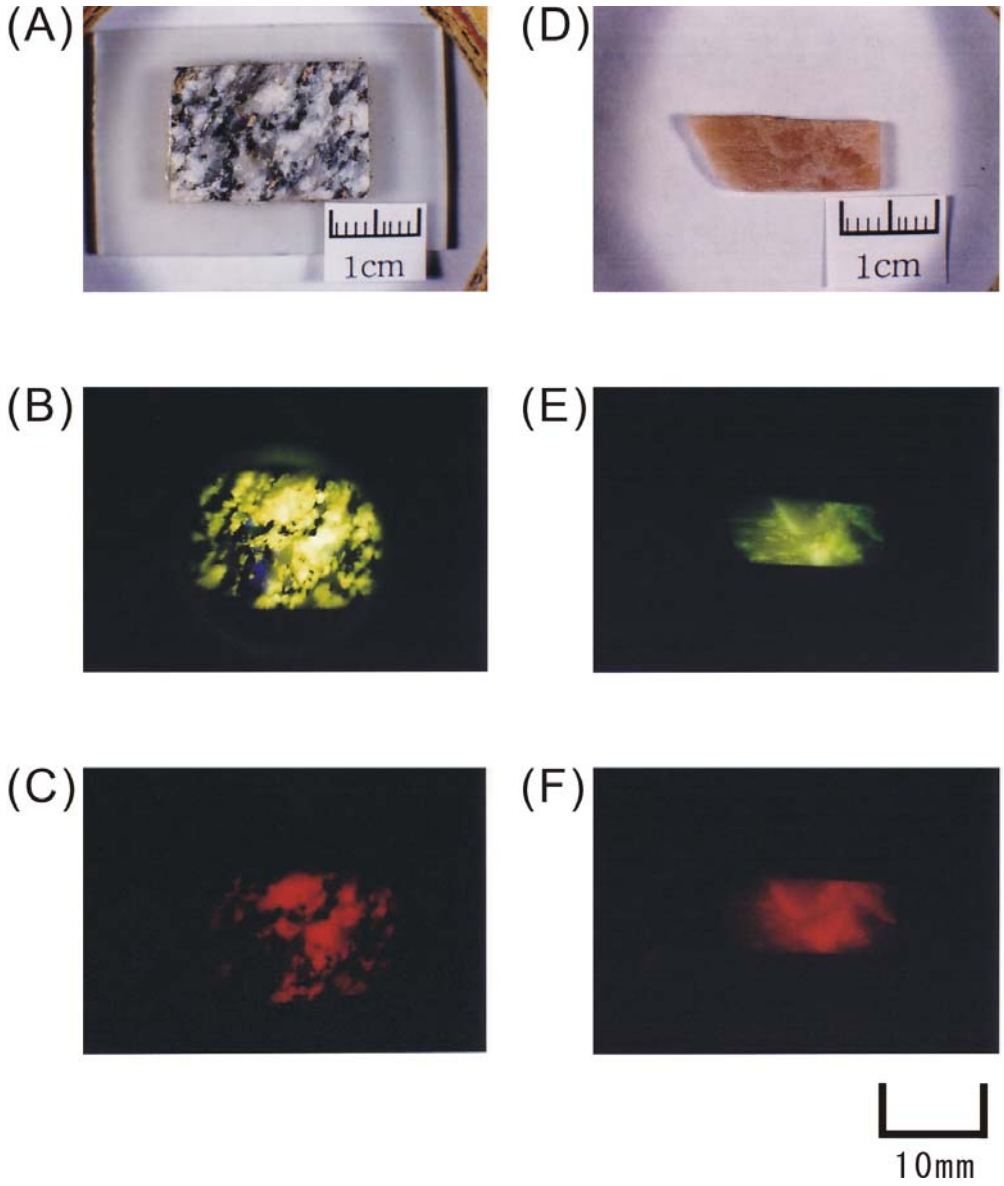

Figure 2.
Typical infrared stimulated luminescence color images (IRSL-CIs) from granite and microcline slices. Real surface images, (A) and (D), are from granite (HW-2) and microcline (India) slices, respectively. Two yellowish IRSL-CIs, (B) and (E), were photographed in visible light regions by interpolating a filter (CF 50E) between camera and slice sample, under illumination by infrared light from LED. Two reddish IRSL-Cis, (C) and (F), were photographed by interpolating an additional filter (R-60). Both slices were irradiated with X-rays to a dose of 200Gy and left for more than one day before photography.

In the granite IRSLCI (B), feldspar (microcline or plagioclase) portions in the real images tend to cause strong yellowish luminescence with IR-illumination, in good agreement with the afterglow and thermoluminescence color images (Hashimoto et al., 1995). There appear faint violet-colored parts, probably due to quartz portions. Although the IRSLCI of granite has been ordinarily observed as either yellow or faint violet (blue), the subsequent experiments proved, with fair certainly, red and faint violet IRSL emission from all feldspar samples. In fact, R-IRSLCI (C) was obtained on the same granite slice by interposing a red filter (Toshiba, R-60) between the sample and camera. Similar IRSLCIs were confirmed on the microcline slice (D-F) and

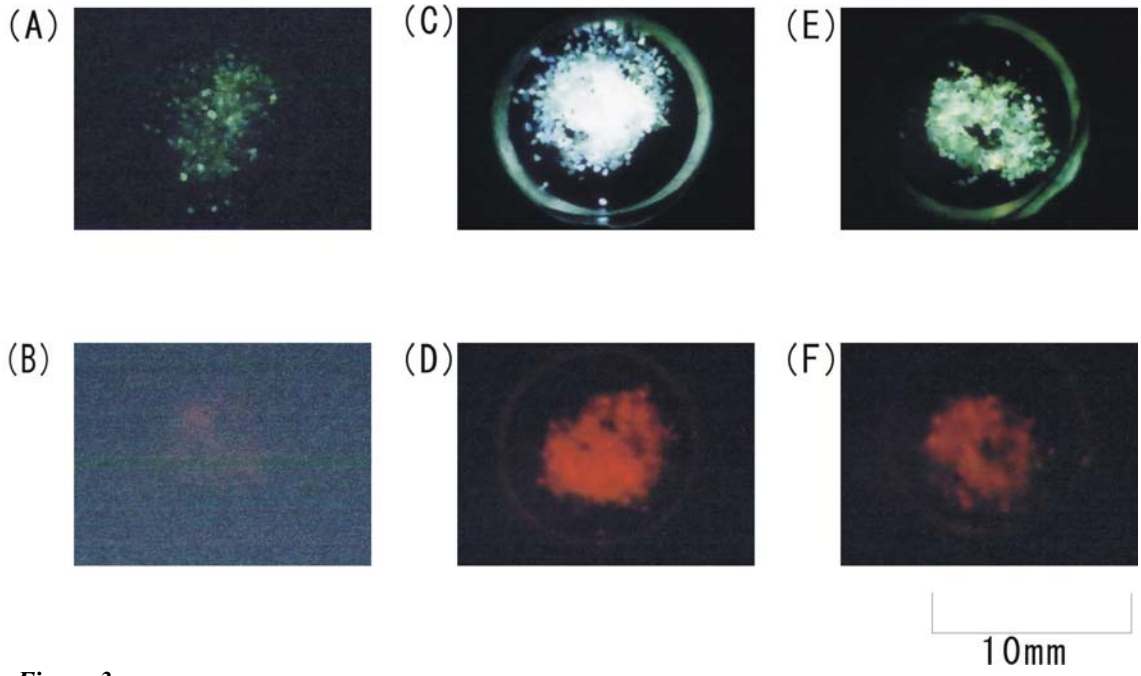

Figure 3.
Dependence of the IRSLCI from albite grain samples on annealing treatments.
Three upper images, (A), (C), (E), and three bottom images, (B), (D), (F), are taken without and with interposing the R-60 filter, respectively. Images (A) and (B) are obtained from as-received albite grains. (C) and (D) images (E) and (F) following thermal annealing treatments in oxidative and reductive condition, respectively, for 3h at 900^{o}C. All samples received of 800Gy of X-rays.

albite grain samples as seen in Figs. 3(A) and (B). Both yellowish and red-IRSL color images show heterogeneous distributions analogous to afterglow and thermoluminescence with feldspar slices (Hashimoto et al., 2001). It should be emphasized here that even natural or as-received samples of both microcline and albite feldspar are emitting R-IRSL

In Hashimoto et al., (1995), albite, potassium and plagioclase feldspar constituents in granite were recognized to be highly sensitive to radiation-induced luminescence, involving radio-luminescence (RL), TL, afterglow, photo-induced phosphorescence as 2-D color images. On the other hand, quartz showed a relatively weak sensitivity to luminescence phenomena even in the color center images and radioluminscence (Hashimoto et al., 2003). The present results support the especially intense luminescence sensitivity of the feldspar portions.

Additionally, it is suggested that light-sensitive trapped electrons (and hole centers) in minerals should greatly depend on the conditions of thermal annealing treatments. The IRSLCIs from albite grain samples, which were annealed with oxidative or reductive conditions, are shown in Fig. 3(C)-(F). It is verified that all of the IRSL-images are enhanced after the annealing treatments, particularly in the R-IRSLCIs. From the annealed sample in oxidative conditions, both visible light in Fig. 3(C) and R-IRSLCIs (D) are enhanced more than the corresponding IRSLCIs in the case of reductive conditions, as indicated in Fig. 3(E) and (F).

2) IRSL-spectra of feldspars

Further quantitative spectral information could be obtained by means of on-line spectrometry. Three spectral results are shown in Fig. 4. Since the luminescence phenomena appear immediately after the IR-LED exposure, the spectra should change from strongest spectrum, which appears in the uppermost spectrum, to weaker ones in the vicinity of final IR-illumination time of 100sec. In the figures the spectra are superimposed upon each other. In agreement with Hashimoto et al. (2002a), the spectra from feldspars consist of a prominent yellowish emission having a peak at 550 nm and orange color having a peak at 580nm. In addition to these peaks; a violet emission emerges at wavelengths shorter than 450nm and another (red) on the long wavelength side beyond 600 nm. The latter emission must correspond to the R-IRSL images as mentioned above in photographs and the spectrum of this is assumed to give a peak at 750nm as expected from afterglow

spectral results (Hashimoto et. al., 2001). Two models of a broad red luminescence around 750nm in feldspars have been suggested: a reaction of the reduced state (Fe(II)) into the oxidation state (Fe(III)) of iron-impurity, alternatively, secondary emission of the Fe(III) excited state, induced by blue photons (Kirsh and Townsend, 1988).

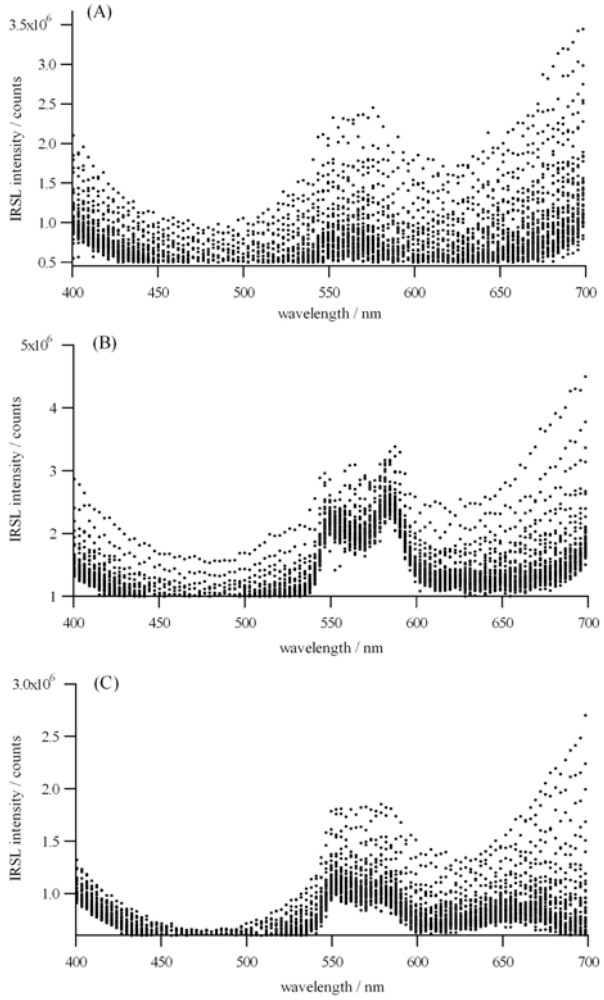

Figure 4.
IRSL-spectra from feldspars by on-line spectrometry with an image intensifier. Every spectrum consists of 100 spectra displaying the decaying behavior of IRSL after start of IR-LED illumination. Feldspar samples are (A) microcline, (B) albite (as-received,) and (C) albite annealed in oxidative conditions. All specimens were exposed to X-rays to doses of 400-800Gy. Each data point consists of photon-counts per 0.1sec interval.

With this in mind, IRSL spectrometry was performed for the thermally annealed albite sample. The spectra are illustrated in Fig.4(C), in which the sample was annealed under oxidative conditions. An enhancement in the red emission regions, by comparison with the yellow emission regions, is clear (c.f. Fig. 4(B)). This result suggests an increase of iron-oxidation state, associated with the oxidative annealing conditions.

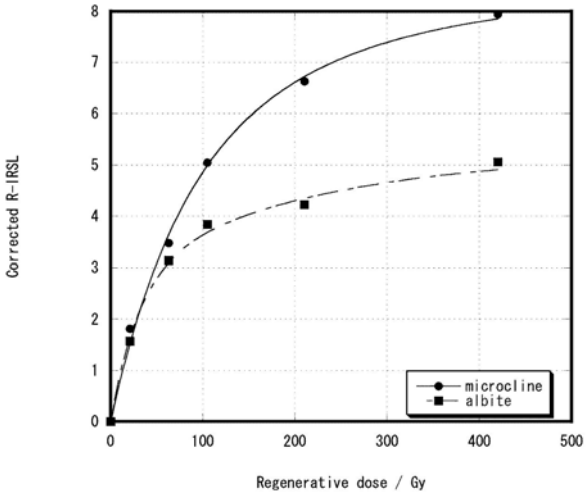

Figure 5.
Growth curves of R-IRSL against additive doses for microcline and albite grains. These two samples were thermally annealed in oxidative conditions. IRSL intensities were integrated for 1 sec of decay curves and corrected by those of the test dose (10.5Gy), of which measurements were carried out between additive dose procedures.

3) Dose response of R-IRSL

To make R-IRSL dose-response curves the R-IRSL values integrated during 1 sec exposure period are plotted in figure 5 against regenerative doses after correction of sensitivity change from IRSL-intensities with the test dose. The final response curves for both microcline and albite samples, which are thermally annealed, are shown. Microcline gives slow saturation trends beyond about 100Gy, whereas albite tends to be saturated rapidly, even at doses less than 100Gy.

This dose-response information of annealed feldspars will be useful for the application of R-IRSL to dating of archaeological porcelain pieces and burnt stones which contain a lot of feldspar constituents, and which might have experienced either oxidative or reductive conditions in their manufacture.

To obtain the optimal measurement of R-IRSL, the detection window widths must be extended into longer wavelength regions by applying the appropriate filter combinations, as well as the reduction of background due to IR-LED as low as possible.

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Reviewer

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