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Evidence of shallow TL peak contributions in infrared radiofluorescence

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HIGHLIGHTS

- Infrared radiofluorescence of K-feldspar was measured.
- A TL-like peak shape is noticeable, after bleaching.
- We demonstrate the presence of a shallow TL trap, responsible for the odd peak.

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ABSTRACT

The notion of what is a 'zero age' is skewed in IR-RF. For bleached sediment, the IR-RF intensities are found to be at their highest values while it falls at its lowest when it reaches dose saturation. During bleaching, it was shown that a sample emits phosphorescence immediately after bleaching, yet very little is known about it. Here, we will try to identify the source for these trapped electrons.

Recent communications in radiofluorescence, have observed an unexpected increase in the beginning of an irradiation, mostly seen in regenerated luminescence. At first glance, this would imply that the increase is tied to an increasingly higher electron capture by the dating trap, during the onset of irradiation. We show that this initial increase is simply due to minute variation in temperature, occurring during the measurements.

1. Introduction

Infrared radiofluorescence (IR-RF) dating of K-feldspar was proposed as a new dating tool by Trautmann et al. (1998), with the characteristic to circumvent anomalous fading. This malign phenomenon leads to severe age underestimation. Very few observations with this technique were carried out since then, due to technical considerations: one must be able to see the fluorescence emitted by feldspar pass through the lead shielding surrounding the beta irradiator. More recently, two luminescence systems have been commercialized, both with the possibility to measure radiofluorescence, a Risø (Buylaert et al., 2012) and a Lexsyg-Research (Richter et al., 2013). With these new systems followed new observations.

In any luminescence measurement, we face the need to regenerate the luminescence signal. Most of the time, this is accomplished by bleaching the samples with light. Regarding infrared radiofluorescence, early accounts report that K-feldspar emitted phosphorescence at room temperature immediately after an optically bleach, at 865 nm (Erfurt and Krbetschek, 2003). Little concern was paid at that time regarding this as it was merely suggested to wait 1 h before regenerating a dose. Another paper will present further details regarding how the IR-RF signal should be reset (Frouin et al., these LED2014 proceedings). Here, we will address the issue of the source of this infrared phosphorescence observed following a bleaching event. In addition, we will elucidate the discrepancy surrounding the recently reported peak shape observed in the beginning of IR-RF measurements.

2. Equipment

All measurements were carried out on a Lexsyg Research device,

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specifically designed for infrared radiofluorescence measurement (Richter et al., 2013). As such, it possesses a ring shaped $^{90}Sr/^{90}Y$ beta source to allow the detection of radioluminescence by positioning a photomultiplier tube directly on top of the aliquot. A near infrared sensitive PMT (Hamamatsu H7421-50), fitted with a thermoelectric cooling system (~- 15 °C) was used in all measurements. The emitted luminescence was filtered through a plano-convex lens and a Chroma D850/40 interference filter (FWHM: 830–870 nm).

In addition, the system has an LED based solar simulator for optically bleaching an aliquot. It is made up of 6 individually controllable bleaching wavelengths: 365 nm (80 mW cm^{-2}), 462 nm (160 mW cm^{-2}), 525 nm (60 mW cm^{-2}), 590 nm (45 mW cm^{-2}), 623 nm (115 mW cm^{-2}) and 850 nm (280 mW cm^{-2}). The K-rich feldspar grains (150–250 μm) were dispensed on 10 mm diameter nickel cups, without making use of silicon oil. In the Lexsyg system, the cup is always in contact with the heating element, at all steps in the measurement cycles. Hence, we choose not to put oil for adherence to prevent staining of the stainless steel foil (25 μm), in place between the sample and the 90Sr/90Y sources. Heating was performed either in nitrogen or in a helium atmosphere, the latter in order to enhance thermal conductivity (Huot, 2007).

Two K-feldspars samples were measured here. One was collected from a Pleistocene marine terrace, in Peru (C5: 76 ± 11 Gy; Pedoja et al., 2006), while the other came from a Gilbert delta in Italy (Bis 4: 680 ± 20 Gy; unpublished data). They were extracted and concentrated using the classical approaches (wet sieving, HCl, heavy liquid separation at 2.58 g/ml), with the addition of a Frantz magnetic separator at the end (Mauz and Lang, 2004). At full strength, the separator helped to remove a lot of iron oxides and other minerals having a weak magnetic susceptibility. Only the “non-magnetic” fraction display luminescence characteristic of feldspar.

3. Observations of a peak shape IR-RF signal

An ordinary radiofluorescence measurement consists of observing directly the phosphorescence emitted by K-feldspar while it absorbs beta radiation. Recently, communications were made on the observations of RF on quartz (Junge et al., 2013; Schmidt et al., 2013) and K-feldspar (Frouin, 2014), which reported an intriguing observation: there is a peak shape in the beginning of the RF measurement (Fig. 1).

The bump in the beginning of the IR-RF measurement is unexpected, as it would imply that an increasingly higher electron trapping rate occurs at the onset of irradiation, followed by a rapid, but slower decrease in trapping. After about 100 s, the IR-RF signal appears to stabilize in a regular pattern of a very slowly decreasing trapping rate, as the trap is being slowly filled with electrons. What is common here and with the measurements reported by Junge et al. (2013) and Schmidt et al. (2013) is that they were all made on Lexsyg Research devices. Also, IR-RF measurements performed on a different system, the Risø TL-DA-20, did not show such a peak (Buylaert et al., 2012), although these authors did report “that we omit the first ~ 20 Gy of data when fitting; this is because we often observe a small increase at the beginning of IR-RF response curves”. In contrast, in all the measurements reported by Trautmann (1999), Erfurt (2003) and colleagues, such an increase was not reported. In that last situation, the measurements had been made on a custom built system, by Daybreak (Erfurt et al., 2003).

3.1. Heating from beneath

We can think of a few hypotheses that may explain this rapid increase in the beginning. First off, let us see if heat is an aspect of it. Why think of heat? Simple, the peak is very noticeable on every Lexsyg Research but not on the other devices. In a Lexsyg Research, the aliquot is always in contact with a heating element. In our measurements, we always controlled the temperature, at 30 °C, slightly above room temperature (~23 °C).

To test the idea, we varied the ramping rate, from room temperature up to 30 °C. The measurement shown in Fig. 1 consisted in raising the temperature (above room temperature), at a constant 5 °C s^{-1} rate, up until 30 °C, and then to immediately begin the IR-RF measurement (Fig. 2).

Other measurements were performed, using either 10 °C s^{-1} or 1 °C s^{-1} ramping rate. The largest signal increase was seen with the highest heating rate. Most likely, here, we are dealing with an issue of thermal lag. Various means can be employed in order to minimize thermal lag (Betts et al., 1993). Here, we already made use of nickel cups, having a very good thermal conductivity. Considering

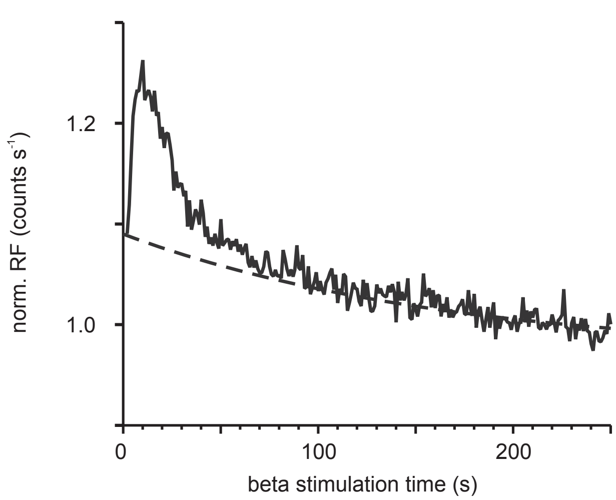

Fig. 1. An IR-RF, performed at a controlled 30 °C temperature. The K-feldspar grains were previously bleached. The dashed curve represents the expected behaviour of IR-RF during irradiation. The beta dose rate is ~0.06 Gy s^{-1} (for a total of 15 Gy, shown here).

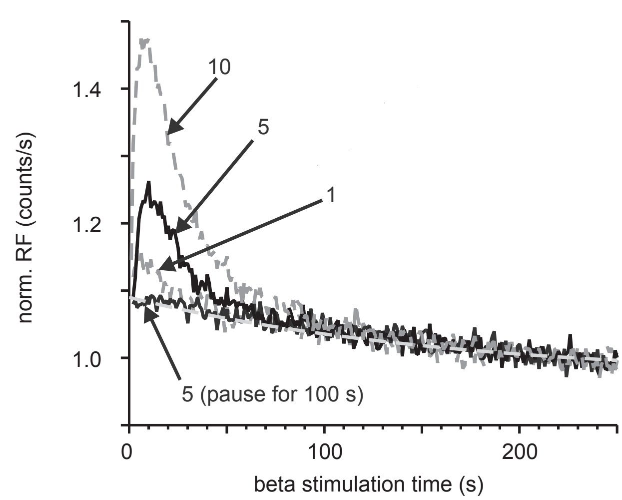

Fig. 2. Repeated IR-RF measurements, where the heating rate (1, 5 or 10 °C s^{-1}) utilized to reach a 30 °C measurement temperature is varied. The aliquot was bleached before irradiating. The light grey dashed curve, overlapping the 100 s paused observation, represents the expected behaviour of IR-RF during irradiation, as in Fig. 1.