Single-grain two-fragment method for dating terrace deposits using red thermoluminescence from quartz

Y. Ganzawa and T. Azuma

Faculty of Environmental Science, Hokkaido University of Education, Hakodate Campus, Hakodate 040-8567, Japan
(e-mail: yganzawa@cc.hokkyodai.ac.jp)

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Abstract
We undertook red thermoluminescence (RTL) dating of terrace deposits in northern Japan using an improved method that requires the analysis of only a single quartz grain. In previous studies, two components (the apparent equivalent dose (Ap-D_e) and the residual level dose (Rl-D_e) after exposure to artificial light) were evaluated from separate multiple-grain aliquots to obtain the age of deposits by RTL and other luminescence techniques.

We developed a new method, called the “single-grain two-fragment” (SGTF) method, to determine the values of Ap-D_e and Rl-D_e. In this method, two fragments are prepared by breaking a single quartz grain using a hammer and nail. The use of recently developed, highly sensitive RTL measurement apparatus enabled the successful detection of RTL signals from single quartz grains (300–500 μm in diameter) with doses as low as 20 Gy. The performance of the SGTF method was assessed by a case study involving the analysis of two samples of terrace deposits. Two separate RTL age groupings (241±20 and 113±17 ka) were determined as minimum equivalent doses (D_e), as deduced from Ap-D_e and Rl-D_e using the single aliquot regeneration (SAR) method. These ages are in good agreement with estimates of the age of the terraces based on geological criteria (OIS 5e and OIS 7).

Introduction

Studies of red thermoluminescence (RTL), based on emissions from 600–650 nm at around 360–390°C from volcanic quartz grains, were pioneered in the 1980s and 1990s (Hashimoto et al., 1986, 1987; Miallier et al., 1991). A series of studies has confirmed the potential of the RTL dating method in evaluating ages over 1 Ma, as trapped electrons are held for periods longer than 10^9 years at ambient temperature (Hashimoto et al., 1987, 1993; Fattahi and Stokes, 2000a). The reliability of these ages is also indicated by the absence of anomalous fading and the excellent RTL reproducibility obtained for repeated irradiations (Fattahi and Stokes, 2003). Using this method, RTL dating has been undertaken using multiple aliquots for volcanic products ranging in age from 2 ka to 1.2 Ma (Fattahi and Stokes, 2003). The advantages of the RTL method also raise the possibility of dating Pleistocene volcanic products and sedimentary deposits.

The application of RTL dating to Pleistocene sedimentary deposits remains limited by problems regarding the bleachability of the RTL signal in quartz. The RTL peak at 360–390°C is not completely bleached under daylight exposure (Miallier et al., 1994; Scholefield and Prescott, 1999; Lai and Murray, 2006). To overcome this problem, previous studies have examined the possible use of other peaks (e.g. 270, 305, and 325°C) because these RTL signals are more rapidly bleached than the 380–390°C peak (Scholefield and Prescott, 1999; Franklin et al., 2000). However, Lai and Murray (2006) reported that residual signals for the peaks at 300–370 and 370–420°C remained at significant levels for quartz from Chinese loess exposed to sunlight for 1260 minutes. To compensate for this residual level, previous studies have evaluated two components (the apparent equivalent dose (Ap-D_e) and the residual level dose (Rl-D_e) after exposure to artificial light) from separate multiple-grain aliquots and obtained the age of the deposits by using the difference between the Ap-D_e and Rl-D_e (Tanaka et al., 1997).

The multiple-grain or multiple-aliquot methods used in previous studies of RTL dating are hampered by another problem; the sample aliquot may contain grains derived from several sources, of different ages, and transported by multiple processes from source areas. The selection of multiple grain aliquots results in different equivalent dose values for each aliquot, depending on the mixture of grains of different origins. To resolve this problem as much as possible, we propose a new dating method based upon the analysis of single grains of quartz. Such an approach is complex since two parameters need to be measured (Ap-D_e and Rl-D_e). Huot and Lamothe (2003) had a similar challenge in their analysis of feldspars, and overcame this by splitting individual grains into two fragments. We have used a similar approach here. Our method is referred to as the single-grain two-fragment (SGTF) method. In this approach, two fragments from the same quartz grain are utilized to obtain the apparent equivalent dose (Ap-D_e) and the residual level dose (Rl-D_e), using the single aliquot regeneration (SAR) method.

Sampling sites and sample preparation

For RTL dating, we analyzed samples NKW-U (NKW-U1, NKW-U2, and NKW-U3) (40°45'39"N, 141°15'40"E) and Fs-U (40°48'0"N, 141°17'58"E) collected from lower and upper terraces upon the Kamikita Plain, located along the Pacific coast of northern Japan (Fig. 1). NKW-U and Fs-U were used for dating by the single-grain two-fragment method (SGTF), and NKW-U1, NKW-U2, and NKW-U3 for multiple-grain dating. The ages of NKW-U and Fs-U are thought to correspond to OIS 7 (186–242 ka) and OIS 5e (127–105 ka), respectively, based on geological criteria (Kuwabara, 2004), comparisons of terrace altitudes, and the ages of two tephra layers (100 and 240 ka) intercalated in the terrace deposits. Both samples contain a reasonable amount of medium-sand-sized quartz grains, presumably supplied from early to middle Pleistocene volcanoes (Hakkodo Volcano and Towada Caldera) located to the west of the sampling sites.

The samples were collected from about 50 cm depth within the selected outcrops to ensure shielding from exposure to daylight. The water content of the samples (water weight/dry sample weight) was measured after drying at 110°C for 1 week. After washing and drying, the grains were sieved into two size fractions (250–350 and 700–900 µm) and treated with 5 M NaOH and 5 M HCl to dissolve glass shards. After magnetic separation, the surfaces of quartz grains were etched with 24% HF for 2 hours to remove glass shards. The purified quartz grains were finally re-sieved into the above two grain-size fractions.

RTL apparatus

RTL emissions from volcanic quartz yield a strong peak at around 360–380°C in the glow curve. To measure weak RTL signals, it is important to completely remove interference by black-body radiation emitted from the heating device at temperatures above 300°C (Fattahi and Stokes, 2000a, b; Stokes and Fattahi, 2003).

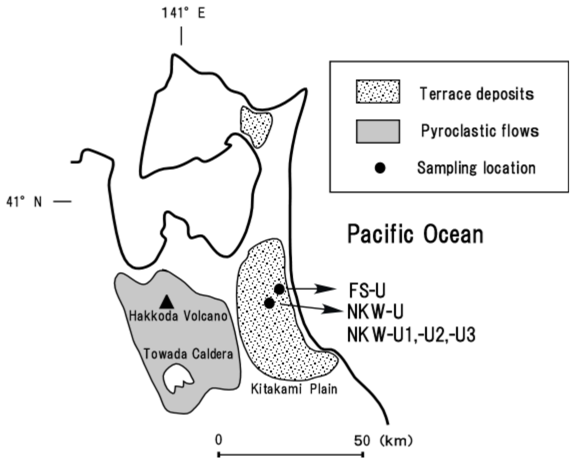

Figure 1: *Location map of the Kamikita Plain, northern Japan, showing the sampling sites for RTL dating.*

Various studies have sought to construct high-performance RTL measurement systems and have examined the optimum combination of filters (Fattahi and Stokes, 2000b; Yawata and Hashimoto, 2004, 2007; Ganzawa et al., 2005; Hashimoto, 2008). We assembled a highly sensitive RTL apparatus equipped with a multi-alkali photomultiplier tube (PMT) (R649s; Hamamatsu Photonics, Hamamatsu city, Japan) encapsulated in a thermoelectric refrigeration chamber (C2761; Hamamatsu Photonics) to minimize the noise signal of the PMT. The heating device, covered by a silver vessel, is able to heat the sample up to 600°C at a rate of 1 or 2°C.s^{-1}. A sample aliquot on a silver plate can also serve to reduce the amount of black-body radiation. The combined use of Hoya O60 (3 mm) and Schott BG39 (3 mm) optical filters enables the optimal detection of RTL at around 600 nm. The use of these components yielded a successful RTL readout, resulting in reliable RTL signals even from a 0.3 mm diameter single grain of quartz. A small X-ray source (VF-50JF; Variant) installed in the present apparatus can supply a dose rate of 6.1 Gy.min^{-1} to the irradiation site at a power of 50 W and 0.1 mA. The use of this X-ray source enables us to apply the SAR method (Murray and Wintle, 2000a, b) as modified by Ganzawa et al. (2005). To eliminate lower-energy X-rays, a 200-µm-thick aluminum absorber was placed in front of the radiation port of the X-ray source (Hashimoto et al., 2002; Hashimoto, 2008). All components of the RTL apparatus were controlled by a personal computer, which enables the successive readout of 10 aliquots for SAR dating.

Prior to the RTL readouts, all quartz grains were screened by 852nm IR laser to eliminate RTL from feldspar inclusions. The preheating condition was set to 220°C for 3 min. All RTL readouts in the experiments were obtained over the range 100 to 450°C at a heating rate of 1°C.s^{-1}. The minimum detection dose of the present RTL apparatus was estimated to be about 20 Gy when analyzing a single grain of 400 µm diameter. For bleaching tests, we used a solar simulator (XC-100B; SERIC) at a power of 100 W. Bleaching of the RTL signal from quartz grains from sample NKW-U reached a stable residual level of approximately 30–50% after an 8-hour bleaching period at a distance of 50 cm from the simulator, corresponding to the power of daylight bleaching for approximately 80–100 hours at a latitude of 45°N.

Experimental methods

1. Multiple-grain method

The multiple-grain method was applied to samples NKW-U1, -U2, and -U3 to evaluate the reproducibility and reliability of the dating method. The resulting ages were then compared with the ages obtained using the SGTF method. The value of D_e for the multiple-grain method was evaluated from two parameters: Ap-D_e determined from an aliquot (approximately 30 grains in each aliquot) using the SAR method, and Rl-D_e was also evaluated by the SAR method after bleaching of quartz grains by the solar simulator (see Figs. 2 and 3). The average Rl-D_e assessed from five aliquots was used for D_e evaluation. Dose response curves were obtained using the integrated signal between 340 and 360°C of the RTL glow curves. Several values of D_e were independently determined from different sets of samples.

2. Single-grain two-fragment method (SGTF)

It is impossible to obtain both Ap-D_e and Rl-D_e from a single grain, because each result must be separately measured using different experimental procedures. To overcome this difficulty, we propose the SGTF method to evaluate Ap-D_e and Rl-D_e individually from two quartz fragments split from a single quartz grain using a hammer and small nail. The SAR method was applied to one of the two quartz fragments to determine Ap-D_e. Rl-D_e was calculated using the SAR method from the second fragment after full bleaching, as employed in the multiple-grain method described above. Details of the SAR experimental method are shown in Fig. 2; regenerative doses were given from 50 to 600 Gy and a test dose of 50 Gy was used. A dose recovery test (204 Gy) was carried out in the final run of the SAR method (Fig. 2). The equivalent dose (D_e) from the original single grain can then be evaluated by subtracting Rl-D_e from Ap-D_e, as calculated independently from the two fragments.

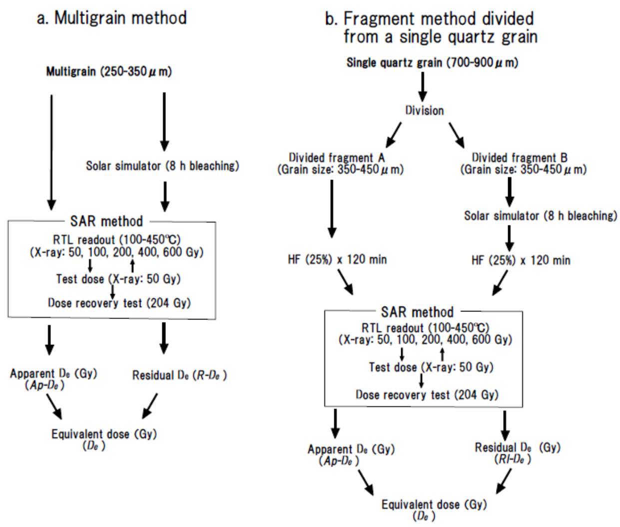

Figure 2: *Experimental procedures followed in determining D_e using (a) the multiple-grain method and (b) the single-grain two-fragment (SGTF) method.*

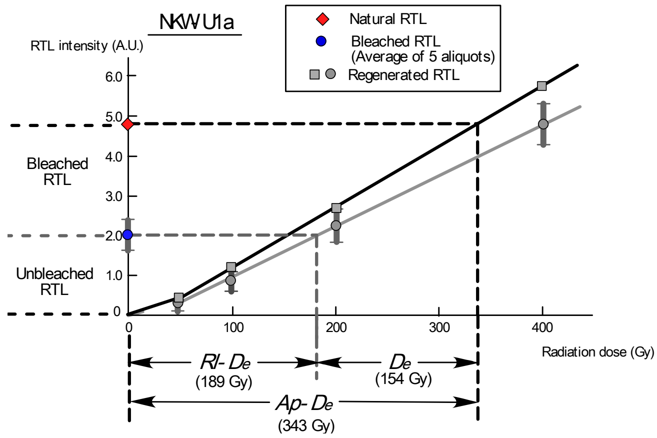

Figure. 3: *D_e evaluation of multi-grain method (Sample code, NKW-U1a; See Fig.2 and Table 1). D_e of an aliquot (30 grains in an aliquot) was determined from the natural sample using the SAR method. Rl-D_e, averaged from five aliquots containing about 30 grains in each, was deduced from bleached RTL intensities after 8-hour bleaching period using the growth curve of NKW-U1a. Ap-D_e was calculated by subtraction of Rl-D_e from D_e.*

3. Annual dose rate

The concentration of uranium and thorium was measured by neutron activation analysis (NAA) at the Inter-University Laboratory for Joint Use of Japan Atomic Energy Research Institute facilities in Japan. An ICP-mass spectrometer was used for analyses of potassium (Ganzawa et al., 2005). The average water

Sample	Ap-D_e (Gy)	Rl-D_e (Gy)	D_e (Gy)	D_a (mGy.a^{-1})	Age (ka)
NKW-U1					
NKW-U1a	343±17	189±19	154±17	0.57±0.1	261±29
NKW-U1b	384±19	212±17	172±16	0.57±0.1	292±28
NKW-U1c	342±14	188±17	154±15	0.57±0.1	261±26
NKW-U1d	356±21	196±14	160±15	0.57±0.1	271±25
NKW-U2					
NKW-U2a	323±16	248±20	75±7	0.42±0.1	169±16
NKW-U2b	303±12	232±26	71±8	0.42±0.1	179±21
NKW-U3					
NKW-U3a	345±17	152±20	193±27	0.50±0.1	386±54
NKW-U3b	332±10	146±23	186±30	0.50±0.1	372±61
NKW-U3c	324±13	143±21	181±28	0.50±0.1	362±56
NKW-U3d	340±20	150±15	190±22	0.50±0.1	380±44

Table 1: *RTL ages calculated using the multiple-grain method. D_e: equivalent dose, Ap-D_e: apparent equivalent dose, Rl-D_e: residual level dose, D_a: annual dose rate, Age: deposit age. Uncertainties in the ages are total errors, including both random and systematic uncertainties.*

content of the samples (18–20 wt%) was measured from three samples collected during three different seasons in a single year. The cosmic doses of 0.11, 0.03 and 0.12 mGy.a^{-1} for NKW-U, NKW-L and FS were calibrated using the standard cosmic ray intensity (0.185 mGy.a^{-1}) at a latitude of 40°N (Prescott and Hutton, 1994). Annual dose rates were estimated from the U, Th, and K contents using conversion data and recently developed conversion factors (Aitken, 1985; Adamiec and Aitken, 1998). Additionally, beta attenuation ratios for 300 µm size quartz grains of 0.81, 0.75 and 0.90 for U, Th and K, were also used for annual dose rates. Thus, the total dose rates at the sampling sites were evaluated to be 0.42–0.57 mGy.a^{-1}.

Results and discussion

1. RTL ages obtained using the multiple-grain method

Analysis of sample NKW-U1 (collected from the uppermost site on the upper terrace) using the multiple-grain method yielded ages in the range 261±29 to 292±28 ka (Table 1). These ages are older than the assigned age of OIS 7 (186–242 ka), as indicated by geological evidence (Kuwabara, 2004). Multiple-grain RTL ages of 169±16 to 179±21 ka and 362±56 to 386±54 ka were determined for NKW-U2 and NKW-U3 at levels located 3 and 5 m below NKW-U1, respectively, again in disagreement with the age of OIS7 (Table 1). These ages are also inconsistent with the known stratigraphy at the sites of the three samples (Kuwabara, 2004).

One possible explanation of the conflicting ages is the mixing in the aliquot of grains derived from several early to middle Pleistocene volcanic sources in the area to the west of the sampling sites (Fig. 1). An additional uncertainty is the bleachability of volcanic quartz, which is strongly resistant against sun-bleaching.

2. Variations in glow curves for single quartz grains

To understand the results from the multiple-grain aliquots, the natural RTL count rate emitted from individual grains was assessed. Figure 4 shows the cumulative sum of the RTL signals from 48 single grains of NKW-U, arranged from brightest to dimmest. The two brightest grains contribute 20% of the total natural RTL counts of the 48 grains, and the nine brightest grains contribute more than 50%, indicating that the value of D_e evaluated using the multiple-grain method is strongly dependent on a small number of the brightest grains.

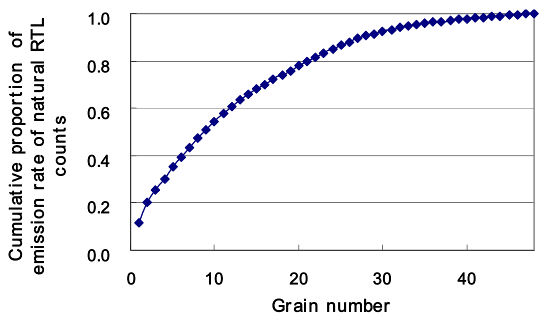

Figure 4: *Cumulative proportion of natural RTL counts for 48 grains. The grains are arranged in the figure from the brightest (grain number 1) to the dimmest (grain number 48).*

We also examined the pattern of RTL glow curves obtained for single grains. Figure 5 shows three representative natural RTL glow curves obtained for single grains of NKW-U, classified into end-member patterns, type-A and type-B, and an intermediate pattern, type-C. Type-A shows a single pronounced peak at around 360°C, whereas type-B and -C show broad patterns, with a mixing of low- and high-temperature (300 and 360°C, respectively) peaks. The different RTL patterns evident in Fig. 5 possibly reflect the diversity of volcanic quartz grains, probably related to magma temperature and component, volcanic age, bleachability and so on. The analytical result of RTL patterns of individual grains showed that NKW-U1 was composed of 11 grains for type-A, 11 grains for Type-B and 2 grains for type-C. The above results indicate that NKW-U consists of various types of quartz grains; consequently, the RTL multiple-grain method is not appropriate for dating this deposit.

3. The effect of hammering on the natural RTL of grains

The SGTF method proposed here involves hitting the single grain with a hammer and nail. This might have affected the RTL signal intensity. Therefore, we assessed the effect of hammering on the natural RTL intensity by examining the SAR D_e and the natural RTL intensity (normalized by a test dose of 50 Gy) in an experiment on a 100 ka (corresponding to 200 Gy) sample of volcanic quartz grains. The effect of hammering was evaluated by comparing the 14 impacted fragments from 7 single grains with 56 impact-free single grains. In Fig. 6 pairs of fragments extracted from the same grain are shown using the same symbol (e.g., gray inverted triangles), and 56 single grains are represented by white diamonds with a blue outline. The fragment pairs show similar D_e values and similar normalized RTL intensities, and values obtained for fragment pairs lie within the field of values obtained for the 56 intact single grains.

The results reveal that the hammering involved in breaking multiple fragments from a single grain using a hammer and small nail results in little change to the original natural RTL intensity of the quartz fragments. Consequently, there was no need to correct the natural RTL intensity for the effect of hitting impact.

4. RTL age of single grains

The effective D_e value required for dating sedimentary deposits can be calculated from SAR measurements of Ap-D_e and Rl-D_e obtained from two quartz fragments from a single grain. All dose recovery tests (204 Gy) showed satisfactory values within a range between 194 Gy and 222 Gy. The value of D_e obtained from many replicate grains by subtracting Rl-D_e from Ap-D_e yields a range, with low values obtained from well-bleached grains and higher values obtained from incompletely bleached grains. In terms of the variation in D_e among grains, the minimum value of D_e is concordant with the effective D_e, providing the best estimate of the age of the deposition; this arises because D_e consists of the pure RTL signal accumulated since the quartz grain was finally fixed in the sedimentary deposit. Radial plot analysis provides an accurate means of showing the range of D_e values.

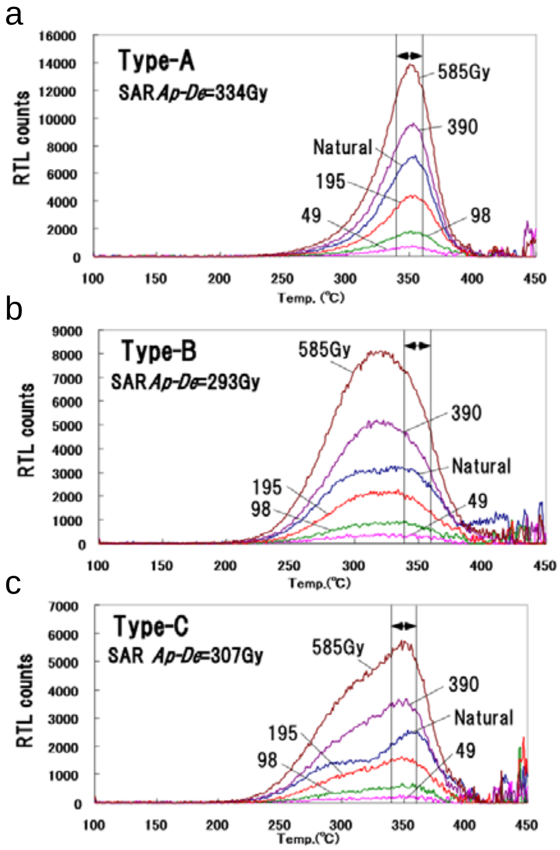

Figure 5: *Three representative glow patterns of the natural RTL of single grains. The obtained patterns were classified as either a mono-peak (a, type-A), a broad peak (b, type-B), or an intermediate double-peak (c, type-C). The RTL intensity was evaluated by the integrated counts for the range 340 to 360℃ in the glow curves (see double-headed arrows). Ap-D_e values evaluated by the SAR method, for regenerated doses of irradiation from 98 to 585 Gy, were 334, 293, and 307 Gy for Type-A, -B, and -C, respectively.*

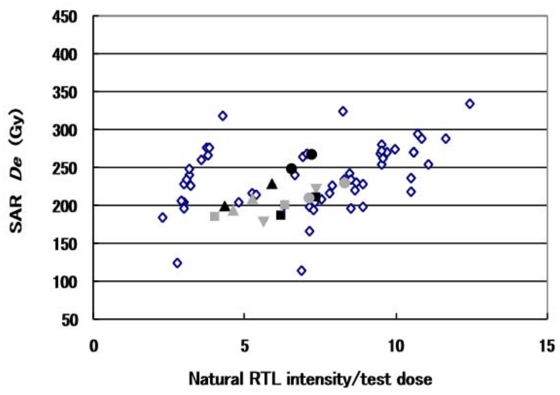

Figure 6: *Comparison of SAR D_e and normalized natural RTL intensity for 14 pairs of quartz fragments derived from individual grains and 56 single grains. The same symbol (e.g., black triangles) is used for each pair of fragments, and the 56 non-divided single grains are represented by white diamonds with blue borders.*

The D_e values obtained for NKW-U1 range from 118 to 408 Gy (Fig. 7a). The eight grains (filled circles) distributed in the 2σ range represent the minimum D_e, and can be regarded as a well-bleached grain mass. The eight grains also passed the χ2-test showing a ratio of 81.4. The RTL pattern of glow curves of these grains within the range of the minimum D_e are composed of three grains of type-A, four grains of type-B and one grain of type -C. This result suggests a wide variation of bleach-process and bleached degree of individual quartz grains. The D_e range of the mass is 118 to 165 Gy, yielding an average age of 241±20 ka based on an annual dose of 0.57 $mGy.a^{-1}$. This age agrees well with the estimated age range of OIS 7 (186–242 ka; Kuwabara, 2004).

In an additional test using sample Fs-U, the D_e distribution also showed a wide range, between 62 to 676 Gy. The average age, evaluated from the minimum D_e of five grains between 62 to 112 Gy in the 2σ range, which showed a ratio of 54.2 for χ2-test, was 113±17 ka based on an annual dose of 0.86 mGy (Fig. 7b). This age is also in agreement with the estimated era of OIS 5 (71–127 ka).

These two ages obtained using the SGTF method demonstrate the validity and applicability of RTL dating of sedimentary deposits based on analyses of single grains of quartz.

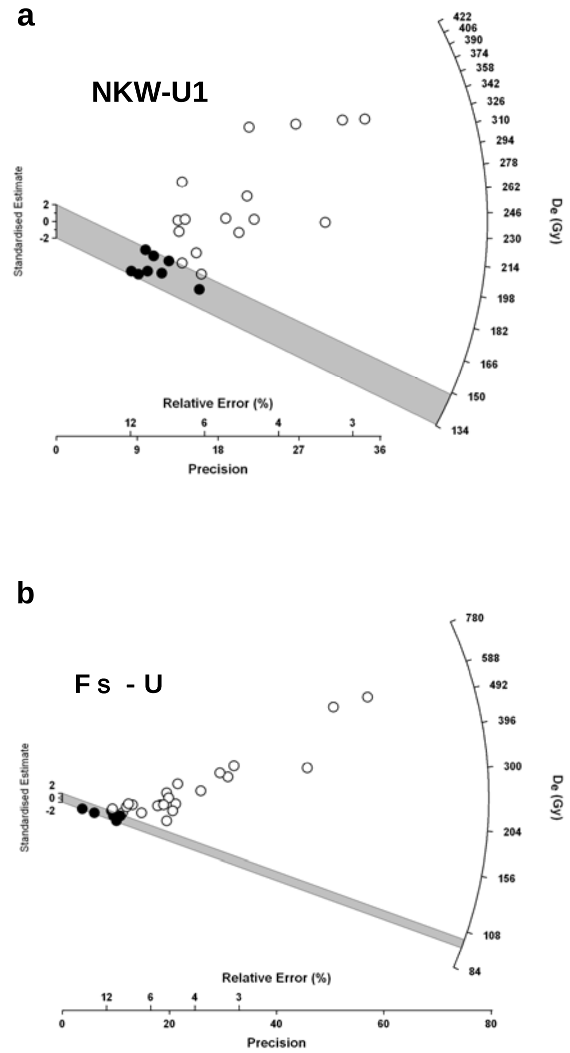

Figure 7: *D_e values of the terrace deposits (corresponding to the deposition age) of NKW-U1 (a) and Fs-U (b), as calculated from divided fragments from single grains (filled circles) within the 2σ range of the radial plot.*

Conclusions

1. We developed an improved RTL apparatus, comprising a multi-alkali PMT, an electric cooling system, and an X-ray irradiator, for the SAR method of dating single quartz grains. The minimum detection performance of this apparatus is about 20 Gy when analyzing a single quartz grain of 0.3 mm in size.

2. An RTL emission study of 48 individual grains revealed that the two brightest grains contribute 20%

of the total natural RTL intensity. This result shows that a small number of the brightest grains dominate the equivalent dose of a deposit when the multi-grain method is applied.

3. The SGTF method was tested based on an analysis of two terrace deposits in northern Japan. The single-grain ages obtained for the two tested samples, distributed within the minimum 2σ range in a D_e radial plot, are in agreement with the geological age and correspond to the timing of transgression in an Oxygen Isotopic stage diagram.

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Reviewer

T. Hashimoto