# THE CHARACTERISTICS OF OSL SIGNAL FROM QUARTZ GRAINS EXTRACTED FROM MODERN SEDIMENTS IN JAPAN

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**Abstract:** Quartz grains from sediments in Japan are derived from complex mixtures of sources, including volcanic, plutonic, metamorphic and sedimentary rocks. We have measured the OSL signal of quartz grains from modern coastal sediments derived from different source rocks and compared these characteristics with the likely source. Each sample shows a different combination of various OSL components. It is concluded that the source rock affects the characteristics of the OSL components from quartz grains in Japanese sediments. By comparing the LM-OSL signals from volcanic sources with those from various source rocks, it can be deduced that quartz which has a higher fast component ratio is more suitable for dating. We also conclude that volcanic source areas should be avoided.

**Keywords:** SAR protocol, LM-OSL, OSL component, Japanese sediments, source rock

## 1. INTRODUCTION

Optically stimulated luminescence (OSL) dating using quartz has been successfully applied mainly to sediments from continental sources, where the quartz has experienced repeated cycles of erosion, transportation and deposition. On the other hand, problems have been reported for quartz originating from active belts (e.g. Indonesia, Italy, Japan, New Zealand and Peru). These include, very low OSL sensitivity and/or no fast component (Preusser *et al*., 2006; Westaway, 2009), age underestimation due to a short lifetime of the signal and anomalous fading (Bonde *et al*., 2001; Steffen *et al*., 2009; Tsukamoto *et al*., 2007).

There have not been many applications of quartz OSL dating to Japanese sediments. Tanaka *et al*. (2001) compared multiple aliquot additive dose (MAAD) and regenerative dose (MAR) protocols for quartz from fluvial terrace sediments; they observed very different equivalent doses ($D_e$) from the two approaches. They also used a single aliquot regenerative dose (SAR) protocol for 48 single quartz grains for one sample and obtained widely scattered $D_e$ values. Some authors have reported that they could not use coarse grained quartz as a dosimeter, because their quartz showed no OSL signal (Kondo *et al*., 2007) or no fast component (Tsukamoto *et al*., 2007). Other authors have focused on loess which mainly originates from the Asian continent (Watanuki *et al*., 2003; 2005; Tsukamoto *et al*., 2003). They found that equivalent dose ($D_e$) values tend to be underestimated due to the presence of the medium and slow 1 components (as defined by Jain *et al*., 2003; Component-2 in Watanuki *et al*., 2005) and assumed that these components originate from more local volcanic quartz. More recently, Tokuyasu *et al*. (in prep) obtained 20-40 ka from 25 quartz samples obtained from shallow marine sediments which had expected ages of 80-130 ka.

The characteristics of the quartz OSL components in Japanese sediments remain poorly known and it is not yet clear that reliable luminescence ages can be derived. It is also important to determine whether the characteristics of the quartz OSL signal depend on the source rock(s) and whether this affects the use of the SAR method. We report here on OSL signals from modern marine and lake sediments derived from different source rocks, and discuss the implications of these observations for dating.

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## 2. SAMPLES

Modern (recently deposited) samples were collected at 6 sites in total. The sediments from 5 sites are each derived from a single source rock type, but sediments from the 6th (Ube) are from a mixture of source rocks. All samples were collected using PVC pipes to prevent light exposure during sampling and were sealed on-site. The material at each end of the pipe, which may have been exposed to light during collection, was discarded in the laboratory.

### Tazawa: volcanic rock source

The Tazawa site is located in the Akita prefecture in the northern part of Honshu (**Fig. 1**). Sand was collected from the Tazawa Lake beach; these sediments are mainly composed of Tertiary volcanic rocks and Tamagawa Welded Tuffs which were erupted between 2 and 1 Ma (Oide *et al*., 1989). Hashimoto *et al*. (1986; 1987) obtained red TL Luminescence Colour Image (TLCI) from the quartz grains from the same site, which suggests the quartz grains are derived from volcanic rocks.



**Fig. 1.** *Location map of sampling sites; (1) Tazawa, (2) Miyajima, (3) Muroto, (4) Kasado, (5) Mukojima, (6) Ube.*

### Miyajima: granite rock source

The Miyajima site is located at the Miya-jima island, Hiroshima Prefecture in the western part of Japan (**Fig. 1**). This island is composed of Hiroshima Granite (~82 Ma; K-Ar ages of biotite; Shibata and Ishihara, 1974). The coastal sand was collected from Hougaura beach in the northern part of the island, and can be reliably assumed to originate almost entirely from the Hiroshima Granite.

### Muroto: sedimentary rock source

This sample was collected from the Ikumi beach located in the Muroto Peninsula, southeast of Shikoku (**Fig. 1**). The Muroto peninsula is located at the southern part of the Shimanto belt and composed mainly of sedimentary rock of including Tertiary sand stone and mudstone (Suyari *et al*., 1991).

### Kasado: metamorphic rock source (low-pressure type)

The sample was collected from the Hanaguri beach, northwest of the Kasado-jima island, Yamaguchi Prefecture (**Fig. 1**). The Kasado-jima island is composed mainly of a low pressure type Cretaceous Ryoke metamorphic rocks, massive biotite granite and magmatitic biotite tonalite. In the northern area, the metamorphic rocks consist mainly of metapelite and metachart, with lesser crystalline limestone and matapsammite (Beppu and Okudaira, 2006). The peak metamorphic conditions for the northern and central area and for the southern area of Kasado-jima island are estimated ~470°C at 200 MPa and ~ 550°C at 250 MPa, respectively (Beppu and Okudaira, 2006).

### Mukojima: metamorphic rock source (high-pressure type)

The sample was collected from the Tanoura beach, southwest of the Muko-jima island, Yamaguchi Prefecture. This island is mainly composed of pelitic rocks of high pressure Sangun metamorphic rocks (~260Ma; K-Ar ages; Nishimura *et al*., 1983), which includes a small amount of sandstone and basic rock. In the northern part of the island, intruded biotite granite rocks are exposed (Sawaki, 1985).

### Ube: mixture

The coastal sand was collected from the beach close to the Maruo port in Ube city, Yamaguchi Prefecture (**Fig. 1**). This sediment is likely to be derived from at least five different types of source rocks from around this site, i.e. Tertiary sedimentary rocks including conglomerate, sandstone and mudstone. Serpentinite from Sangun metamorphic rocks and granites are also exposed (Igi *et al*., 1987).

All the samples were prepared by first sieving to between 75-125 μm, treated with 36% hydrochloric acid for 48 hours and with 20% sodium hydroxide for 24 hours. The density fraction between 2.63-2.68 g·cm$^{-3}$ was then separated using sodium polytungstate solution, and etched with 23% hydrofluoric acid for 2 hours and with 20% hydrofluorosilicic acid over 7 days. The calibration quartz provided by Risø-DTU was also measured for comparison; it is known to be dominated by the fast component (Jain *et al*., 2008), the most suitable component for dating sediments when using the SAR protocol (Murray and Wintle, 2006).
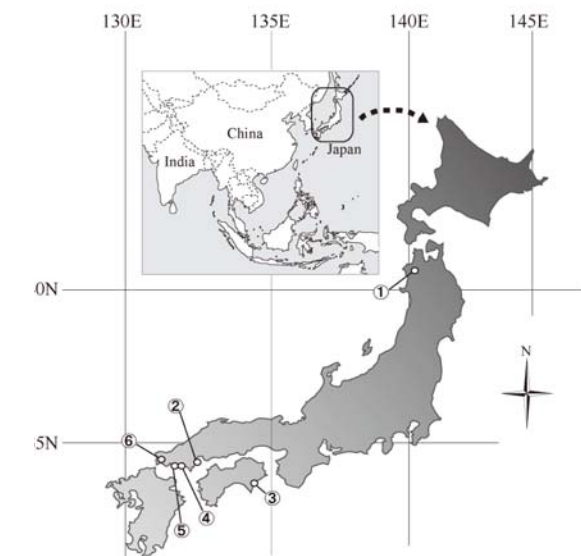
## 3. INSTRUMENT AND OSL MEASUREMENTS

All luminescence measurements were made with a Risø TL/OSL-DA-15 system (Bøtter-Jensen *et al.*, 2003) equipped with a calibrated $^{90}$Sr/$^{90}$Y beta source (~0.1 Gy/s). Blue light emitting diodes (LEDs) were used for stimulation, and the luminescence signal was detected in the ultraviolet (UV) region through a 7.5 mm HOYA U-340 filter. All luminescence measurements were carried out on coarse grained quartz (75-125 μm) using preheats between 160 and 300°C (10 s) and blue light stimulation at 125°C for 40 s with the single aliquot regenerative-dose (SAR) procedure (Murray and Wintle, 2000), which is shown in **Table 1**. Cut-heat was set to 40°C below the preheat temperatures (from 120 to 260°C). At the end of each measurement cycle a blue light stimulation at 280°C for 40 s was conducted to prevent signal carry over to the next cycle. At the end of the SAR sequence, the same regeneration dose as the first regeneration dose (22 Gy) was repeated to check whether the sensitivity corrected

**Table 1.** *Experimental flow chart for De estimation and LM-OSL measurement.*

| Step | Treatment |
|---|---|
| 1 | Give dose (0 Gy if natural) |
| 2 | Preheat at 160-300°C for 10 s |
| 3 | OSL measurement at 125°C for 40 s → $L_x$ |
| 4 | Give test dose |
| 5 | Cut heat at 120-260°C for 10 s |
| 6 | OSL measurement at 125°C for 40 s → $T_x$ |
| 7 | Hot bleach by Blue-LED at 280°C for 40 s |
| 8 | Repeat from step1 to 7 |
| 9 | Give dose to 88 Gy |
| 10 | Preheat at 260°C for 10 s |
| 11 | LM-OSL measurement (125°C for 1000 s) |

OSL ($L_x/T_x$) was reproducible (recycling ratio). After a conventional SAR cycle, Linear Modulated (LM) OSL was measured by linearly increasing the power of the LEDs from 0 to ~78 mWcm$^{-2}$. LM-OSL measurements were made for 1000 s at 125°C after irradiating the aliquots with 88 Gy and preheated at 260°C for 10 s. The resulting LM-OSL curve was fitted with the following
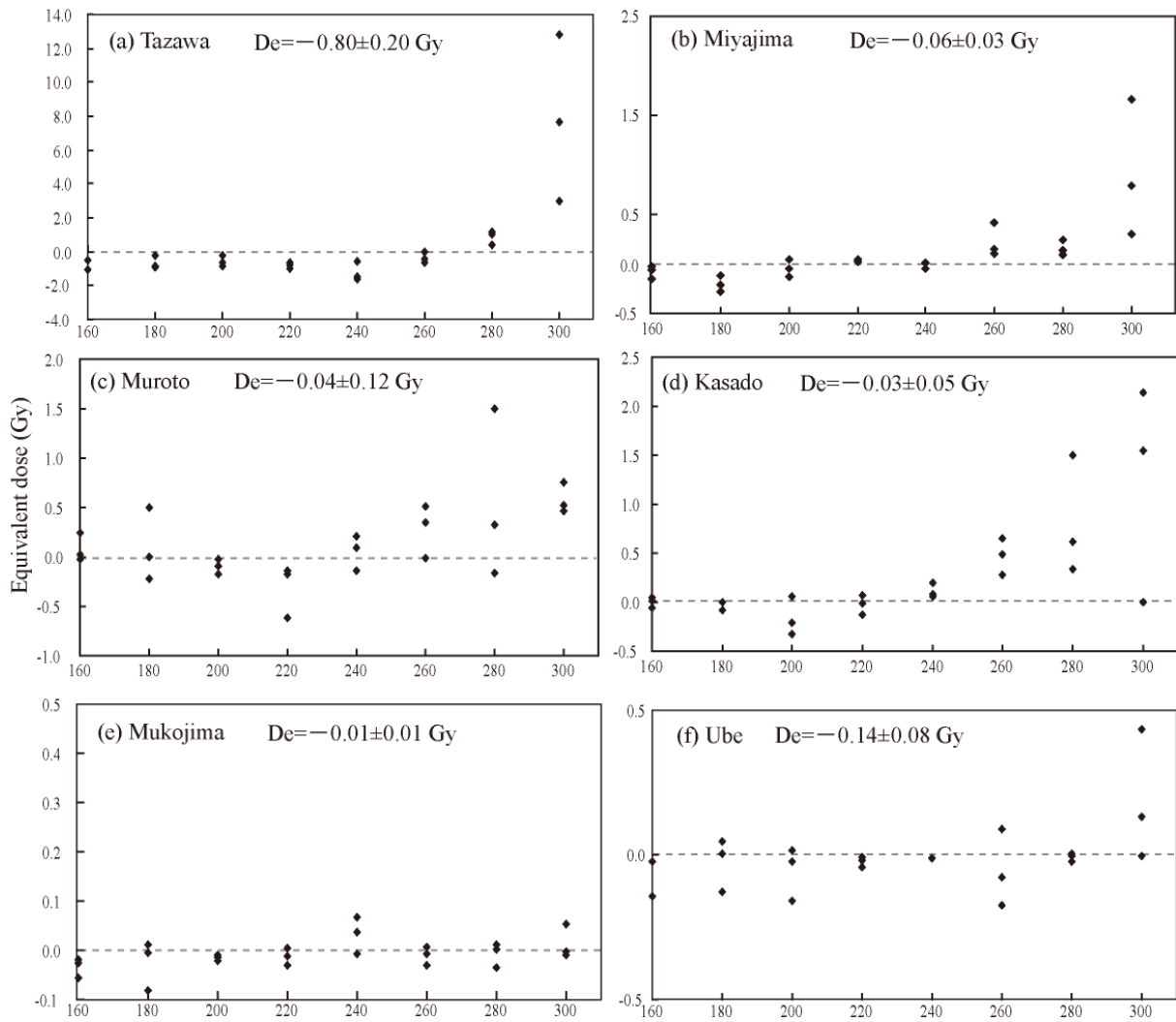


**Fig. 2.** *$D_e$ estimates (n=24) as a function of preheat temperature. The average $D_e$ (n=15) given on each diagram is based on the temperature range 160 to 240°C.*

equation to separate into $i$ different components:

$$L(t) = \sum A_i b_i \left( \frac{t}{P} \right) \exp\left( \frac{b_i t^2}{2P} \right), \qquad (3.1)$$

where $A_i$ is the amplitude, $b_i$ is the detrapping probability (proportional to photoionization cross section $\sigma_i$) and the maximum light intensity $I_0$ ($b_i = \sigma_i I_0$), $P$ is the total observation time and $L(t)$ is the OSL signal at time $t$ (Bulur, 1996).

## 4. OSL CHARACTERISTICS OF QUARTZ GRAINS FROM SEDIMENTS IN JAPAN

The $D_e$ values of the modern beach sediments were obtained using the SAR protocol with different preheat temperatures between 160 and 300°C. **Fig. 2** shows the $D_e$ values at different preheat temperatures. At each temperature, 3 aliquots were used; for $D_e$ estimation, the first 0.8 s of stimulation was integrated, and the 0.96-1.76 s integral subtracted as background. All the $D_e$ values are close to zero for preheat temperatures between 160-240°C, indicating that the quartz grains were well bleached (see **Fig. 2** and **Table 2**). However, the $D_e$ values increase for preheats above 260°C. This increase can be explained by thermal transfer (Aitken, 1998). **Fig. 3**

shows the sensitivity change through the SAR cycles ($T_i/T_n$) for all samples using 6 aliquots, for 260°C preheats. The sensitivity of the OSL signal increased by 2-3 times during the SAR cycles for the samples from volcanic rock source (Tazawa), granite rock source (Miyajima), high-pressure metamorphic rock source (Mukojima) and low-pressure metamorphic rock source (Kasado), however those samples from sedimentary rock
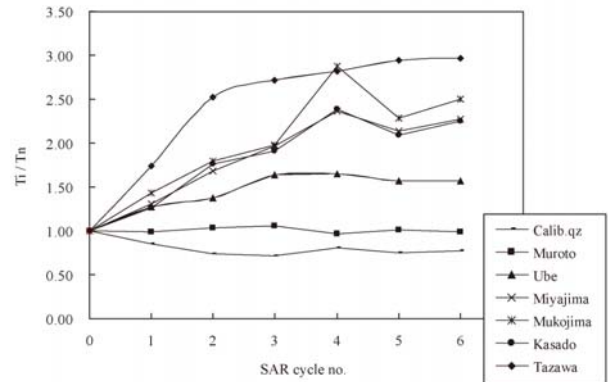


**Fig. 3.** $T_i/T_n$ ratio preheated at 260°C for 10 s as a function of the SAR cycle.

**Table 2.** *Results of experimental measurements. The $D_e$ values are estimated average from 160 to 240 °C preheat temperature (n=15).*

| Sample | Source | $D_e$ (160-240°C, Gy) | Dose recovery ratio | Recycling ratio | R0 /RN (%) |
|---|---|---|---|---|---|
| Tazawa | volcanic | -0.80±0.20 | 0.98±0.14 | 0.81±0.13 | 155 |
| Miyajima | granitic | -0.06±0.03 | 0.91±0.01 | 1.12±0.05 | 21 |
| Muroto | sedimentary | -0.04±0.12 | 0.92±0.04 | 0.97±0.03 | 54 |
| Kasado | metamorphic | -0.03±0.05 | 1.00±0.18 | 1.03±0.04 | 6 |
| Mukojima | metamorphic | -0.01±0.01 | 0.98±0.01 | 1.17±0.05 | 210 |
| Ube | mixed | -0.14±0.08 | 1.03±0.13 | 0.98±0.01 | -65 |

**Table 3.** *Summary of OSL components of quartz grains from sediments in Japan.*

| Sample | OSL components | Detrapping probability (s⁻¹) | Photoionisation Cross Section (10⁻¹⁸ cm²) | Relative Cross Section to fast component | Relative Intensity to fast component |
|---|---|---|---|---|---|
| Calib. Qz | Fast | 6.1 | 32.8 | 1 | 1 |
| | Medium | 1.2 | 6.4 | 0.20 | 0.26 |
| Tazawa | Medium | 1.4 | 7.5 | 0.31 | — |
| | Slow1 | 0. | 2.3 | 0.09 | — |
| Miyajima | Fast | 4.9 | 26.3 | 1 | 1 |
| | Medium | 1.0 | 5.4 | 0.21 | 1.05 |
| Muroto | Fast | 3.3 | 17.6 | 1 | 1 |
| | Medium | 0.7 | 3.9 | 0.22 | 1.30 |
| Kasado | Fast | 5.5 | 29.4 | 1 | 1 |
| | Slow 1 | 0.4 | 2.2 | 0.07 | 0.43 |
| Mukojima | Fast | 4.3 | 23.4 | 1 | 1 |
| | Medium | 0.9 | 4.8 | 0.20 | 2.00 |
| Ube | Fast | 6.3 | 33.9 | 1 | 1 |
| | Medium | 1.6 | 8.75 | 0.26 | 0.82 |
| Jain et al. (2003) | Fast | — | 23.2 | 1 | — |
| | Medium | — | 5.6 | 0.24 | — |
| | Slow 1 | — | 1.3 | 0.06 | — |
| Singarayer and Bailey (2003) | Fast | — | 25.0 | 1.00 | — |
| | Medium | — | 5.9 | 0.24 | — |

source (Muroto) and mixture of source rocks including sedimentary rock (Ube) showed no significant sensitivity change. This may suggest that the quartz grains from sedimentary rock have already been sensitised. The recycling ratio $(L_5/T_5)/(L_1/T_1)$ is satisfactory in 4 out of 6 samples, suggesting the sensitivity correction using a test dose is performing well in the cases. However, the recy-

cling ratios from volcanic rock source and high-pressure metamorphic rock source, of 0.81±0.13 and 1.17±0.05, respectively, are not so satisfactory. $R_0/R_N$ ratios from the 2 samples are 155% and 210%, respectively, indicating a much higher level of recuperation in these samples (**Table 2**).
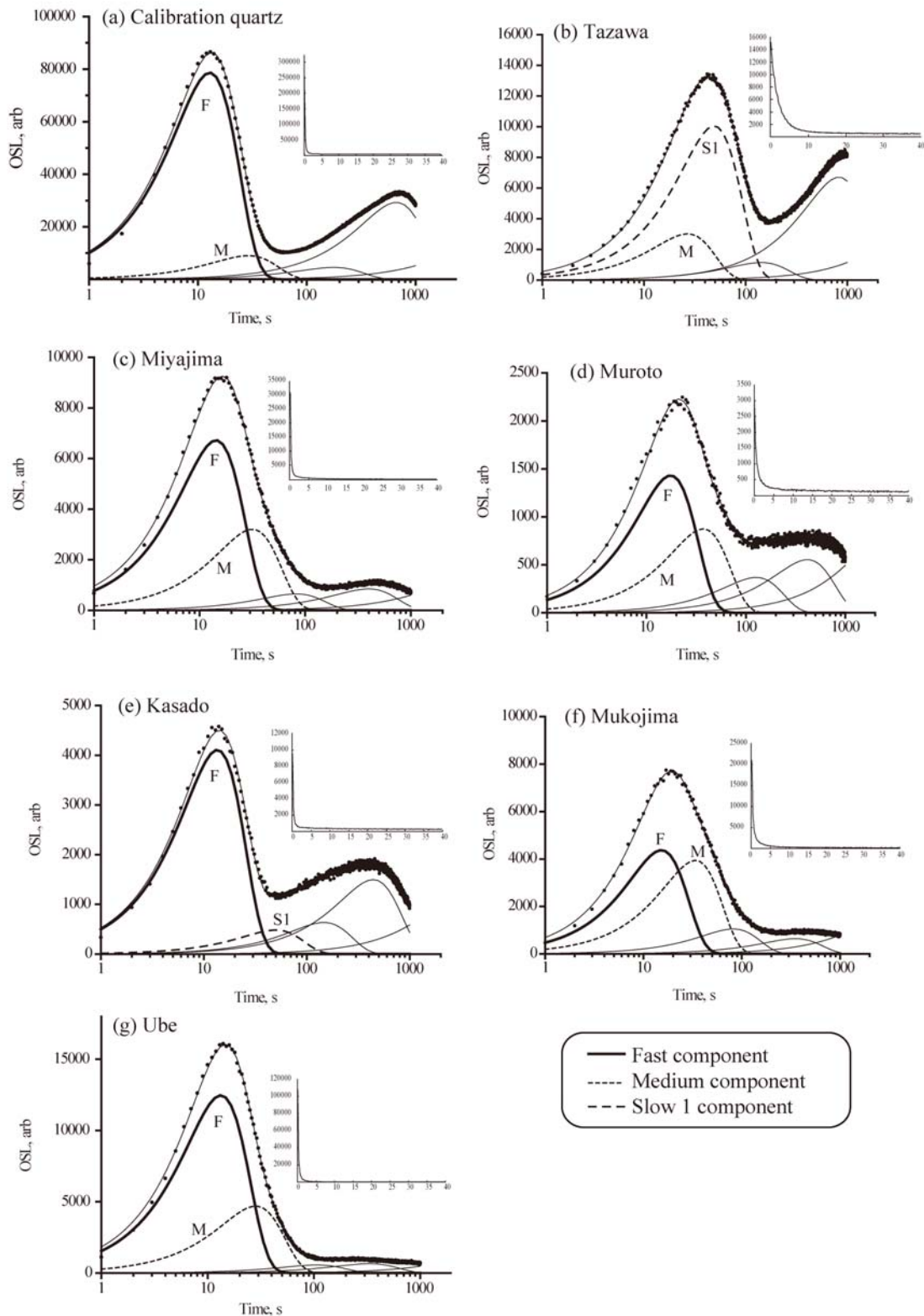


**Fig. 4.** *LM-OSL signal from 88 Gy irradiated aliquots, after a preheat to 260°C for 10 s. Inset shows the decay curve of 22 Gy irradiated aliquot of each sample, after the same preheat.*

A dose recovery test was conducted for all samples using 3 aliquots for Miyajima and Ube samples, and 6 aliquots for other samples. The aliquots were bleached with blue light without heating, and then given a beta dose of 33 Gy. The SAR protocol was applied, using a 260°C preheat, to test whether the given dose was successfully recovered. All samples, including those from Tayawa and Mukojima, gave a recovered/given dose ratio close to unity (within ± 10%; **Table 2**), showing that our SAR protocol is able to determine a dose given to these samples before any heating.

**Fig. 4** shows the LM-OSL curves of the quartz grains from Risø calibration quartz and the 6 modern Japanese samples. The LM-OSL signals were fitted with between 4 and 6 components (usually fast, medium and 2-4 slow components). A summary of the results of this fitting process is shown in **Table 3**. Each sample shows a different combination of various OSL components. In particular, the sample from the volcanic source area does not have a detectable fast component but only a medium and a strong slow-1 component (using the definition of Jain *et al*., 2003). As a result, the continuous wave (CW) OSL curve of this sample shows a slower decay compared to that of calibration quartz (**Fig. 4**), and significant thermal transfer at higher preheat temperatures above 260°C (**Fig. 2**). According to Tsukamoto *et al*. (2007) these luminescence characteristics are typical of volcanic quartz; they also found that the OSL signal from volcanic quartz fades. The fast component is present in all our other samples, although all have a larger relative medium component contribution compared to calibration quartz. The low pressure metamorphic sample is an exception to this; it has no detectable medium component. It is interesting to note that in this sample one can expect that the fast component was sensitised during the metamorphic process (Tsukamoto *et al*., 2010). Poor recycling ratios, 0.81±0.13 and 1.17±0.05 were obtained from the Tazawa and Mukojima samples, respectively; these two samples have the smallest relative contribution of the fast component (**Table 3**). All the other samples show a relatively strong fast component, and these are the samples which show satisfactory dose recovery and recycling ratios.

## 5. CONCLUSION

This paper compares the OSL components of quartz grains derived from different source rock areas: volcanic, granitic, metamorphic (both high and low pressure type), sedimentary and mixed. Each sample shows a different combination of the various OSL components and this resulted in different performance in the SAR protocol. The quartz samples which showed dominant fast component (Ube and Kasado) showed best recycling and dose recovery ratios, which suggests these samples are suitable for OSL dating using SAR. On the other hand, the samples with no or only a small fast component (volcanic and high pressure metamorphic source) showed good dose recovery ratio but a poor recycling ratio and greater sensitivity changes; these samples are unlikely to be suitable for OSL dating.

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