

## Potential of optically-stimulated luminescence (OSL) dating to improve Quaternary geology in Japan

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The optically-stimulated luminescence (OSL) dating has become a versatile and common method for determining depositional ages of Late Quaternary sediments worldwide over the last 15 years like the radiocarbon dating. The major advantage of this method, compared to the radiocarbon dating, is its applicability to ubiquitous minerals including quartz and feldspar in a wider age range, from several decades to a million years. Its disadvantage in contrast is problems in accuracy and reliability that are derived from uncertainties in the dose rate during burial and from regional variations in luminescence properties of mineral grains. The OSL dating is therefore regarded as an alternative, but still very effective, method where the radiocarbon dating does not work for some reasons. On the contrary to the recent expansion worldwide, the application of the OSL dating to sediments in Japan has been limited yet. The main reasons for this include unfavorable luminescence properties of quartz, especially of volcanic origin, and less importance of the OSL dating due to well-established Late Quaternary tephrochronology in Japan. However, some modified or even standard protocols of the OSL dating, especially those using K-feldspar, have been proved to effective for Japanese sediments, and there are still many cases in which the OSL dating is expected to have a critical role. We review the recent application of the OSL dating to Japanese coastal sediments, including in coastal dunes, beach ridges, tsunami deposits, raised marine terraces, and alternations of loess and tephra, and summarize their achievements and problems.

Keywords: Quaternary, geology, coast, luminescence dating

## Optically stimulated luminescence dating of marine sediments: a review

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Optically stimulated luminescence (OSL) dating determines the time that has elapsed since sediments were last exposed to sunlight; the technique is widely applicable to late Quaternary sediments. An OSL age is calculated by dividing the radiation dose that has been absorbed by mineral grains during burial by the rate of energy absorption from ionizing radiation during burial (dose rate). The dose is measured using luminescence techniques and the dose rate is calculated from a knowledge of the concentration of natural radionuclides (U- and Th-series and <sup>40</sup>K) in the sediment matrix. A prerequisite for obtaining an accurate luminescence age is that all grains have been exposed to sufficient sunlight to empty the prior trapped charge prior to burial.

In contrast to studies on land, the application of OSL dating to marine sediments has been limited. The main reasons appear to be difficulties during sample collection (ensuring light shielded condition), the usually fine-grained nature of the material and the evaluation of the life-time burial water content. Here we give an overview of luminescence dating applied to marine cores, discuss the recent methodological advances and the upper and lower age limits. Finally, we show the potential of OSL dating of marine cores when high sampling depth resolution is available.

Keywords: optically stimulated luminescence dating, Quaternary

## Luminescence dating of last Pleistocene marine terrace

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The marine terrace formed during sea-level highstand is important to understand the tectonics in the coastal area, Japan. To estimate the formed age of marine terrace is a key to reconstruct the uplift rate which is one of the most important tectonic information. In previous works, there were some uncertainties depending on indirectly age determination based on tephrochronology of terrestrial sediment, distribution of marine terraces in that area and so on. To determine the age of marine terrace directly, Post-infrared infrared stimulated luminescence (pIRIR) dating which can be used for sand samples from Late Pleistocene to present, was applied to the marine terrace. On the other hand, pIRIR signal was also applied to modern beach sands to know the difference of signal stabilities between difference areas and residual dose which was one of the most important factors in pIRIR dating. In seven outcrops of marine terraces of MIS5e, 7, 9 and 11 at Kamikita coastal plain, pIRIR ages were determined by using K-rich feldspar from subtidal sediments. Each age had no age difference between each subtidal facies of same outcrops because of measurement precision. However, considering error range, the average ages of samples from all subtidal facies of same outcrops were relatively concordant with expected ages of marine terrace which were based on Koike and Machida (2001). On the other hand, the luminescence characteristics of pIRIR signal such as signal stability were difference between each sampling area in Japan. This difference influenced the suitable choice of pIRIR protocols which had several differences between measurement temperatures. As a result, it suggested that we could apply pIRIR protocols to other marine terraces in Japan too, considering the signal stability depending on sampling area.

\*This research project has been conducted as the regulatory supporting research funded by the Secretariat of the Nuclear Regulation Authority, Japan.

Koike K. and Machida H., 2001. Atlas of Quaternary Marine Terraces in the Japanese Islands. University of Tokyo Press, ISBN 4130607359 (in Japanese).

Keywords: pIRIR dating, Marine terrace, Late Pleistocene

## Luminescence chronology of the Middle Pleistocene marine and fluvial terraces in northern Japan using pIRIR dating

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In northern Japan, it has been difficult to construct a detailed chronology of marine / fluvial terraces of the Middle Pleistocene, due to the lack of the marker tephra layers and to the deformation of original landforms by strong past periglaciations. The lack of age constraint has prevented studies of precise geomorphic development and palaeoenvironmental reconstruction in this area. This study applies a post-IR IRSL (pIRIR;Thomsen et al., 2008; Buylaert et al., 2009) SAR protocol using polymineral fine grains to marine and fluvial terraces at Tonbetsu plain and Gifu Terraces along the Sea of Okhotsk coast area in northern Hokkaido, at Setana plain in southeastern Hokkaido.

In Tonbetsu plain, northern Hokkaido, the pIRIR ages from the higher marine terraces are ca.340 -370 ka, which yielded ages corresponding to MIS 9, respectively. In Setana plain, in southeastern Hokkaido, the pIRIR ages from the Oyachi marine terraces are MIS 7. In addition, pIRIR ages from upper Setana formation, basement of Tonke-gawa fluvial terraces, are ca. 400 ka. These pIRIR ages indicate that upper limit age of Setana formation and development of landforms after the Middle Pleistocene in Setana plain. I will also introduce pIRIR dating results and its meanings of marine / fluvial terraces of the Middle Pleistocene, northern Honshu Island.

### Reference

Buylaert et al., (2009) *Radiat. Meas.* 44, 560-565.; Thomsen et al.(2008) *Radiation Measurements*, 43, 1474-1486.

Keywords: pIRIR dating, northern Japan, marine terrace, fluvial terrace, Middle Pleistocene

## Potential and problems of K-feldspar optically stimulated luminescence dating of tsunami deposits

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In active tectonic region (e.g. the Japanese islands), quartz is often not suitable for optically stimulated luminescence (OSL) dating because of a lack of fast component and/or fading. Although OSL dating of feldspar is also disturbed due to fading and necessity of longer sunlight exposure for bleaching, stronger infrared stimulated luminescence (IRSL) relative to quartz OSL accepts for single grain OSL measurements and dating of young samples. This study was undertaken in order to confirm potentials and problems of OSL dating of K-feldspar on tsunami deposits using the 2011 Tohoku-oki tsunami deposits at Soma and Minami-soma city and Jogan tsunami deposits (A.D. 869) at Minami-soma city, Fukushima Prefecture, northeastern Japan.

Equivalent dose ratio of the conventional IRSL signal ( $IR_{50}$ ) and post-IR IRSL signal ( $pIRIR_{150}$ ) indicated both the 2011 Tohoku-oki and Jogan tsunami deposits were probably unexposed sunlight during transport processes. Although this condition was not favorable for OSL dating, single grain K-feldspar IRSL measurements of the 2011 Tohoku-oki tsunami deposits were feasible to extract the bleached grains indicating true depositional age. It is interpreted the 2011 Tohoku-oki tsunami deposits include K-feldspar grains bleached just before the tsunami transportations. On the other hand, single grain K-feldspar IRSL measurements of the Jogan tsunami deposits were only able to extract one grain showing true depositional age out of the 199 grains in five samples, and about half of the equivalent doses of K-feldspar grains exceeded 200 Gy (saturation level). The results indicated main sediment sources of Jogan tsunami deposits were different from the 2011 tsunami deposits at Minami-soma city, and therefore it caused different amounts of the K-feldspar grains bleached just before the tsunami transportations.

This study indicates it is important for estimating accurate depositional age of tsunami deposits using OSL dating to consider sediment sources and to select grains which have received sufficient sunlight exposure just before tsunami transportations.

Keywords: optically stimulated luminescence dating, post-IR IRSL dating, single grain IRSL dating, K-feldspar, tsunami deposits, Fukushima Prefecture

## Journey of sand grains from river to deep marine estimated from bleaching percentage (BLP) of feldspar grains

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Optically stimulated luminescence has two remarkable properties, (1) the intensity of mineral grain luminescence increases with the amount of ionizing radiation absorbed by the grain; and (2) sufficient exposure to sunlight results in resetting of the luminescence signal (bleaching). Using these properties, evaluation for sand grain transport with the content of completely bleached grains (bleaching percentage: BLP) was established. Several case studies from riverine sand (Shirai et al., 2008; Shirai and Hayashizaki, 2013) to deep marine turbidite sand (Shirai and Hayashizaki, 2013) and sand grain transport processes inferred from these results are introduced.

Keywords: IRSL, bleaching, transport process

## Red thermoluminescence (RTL) dating of Pleistocene volcanic quartz

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The Red thermoluminescence (RTL) method using tephra quartz has been widely used to date Pleistocene volcanic activity. The RTL method has some useful advantages explained as follows; long trap life time (more than  $10^7$ - $10^9$  years at room temperature 300K), little effect of anomalous fading and exceeding ability of dose response (Yawata and Hashimoto, 2004). The great potential of RTL method is realized when it is used for the dating of tephra products covering Pleistocene land forms in Japan. Additionally, single quartz grain RTL dating using SAR method (Wintle and Murray, 2003) was also employed for late Pleistocene tephra and succeeded in giving more accurate ages (Ogawa et al., 2011).

RTL research we have been carrying out using some tephra quartz grains in middle Pleistocene (ex. Hakkoda 1<sup>st</sup> stage pyroclastic flow) showed three type RTL emission patterns; mono-peak type, double-peak type and broad type.

A broad type with double peaks at around 300 and 360°C showed a quite different pattern when compared to the typical shape of a mono-peak type being commonly used for RTL dating, such as a late Pleistocene Toya tephra (Ganzawa et al, 2005).

The broad type of RTL emission is originating in ignimbrite, suggesting a high emplacement temperature over 800°C. Heating quartz grains up to 1000°C, using Hakkoda aquatic pyroclastic flow fixed at a temperature lower than 200°C, clearly showed a change of the RTL emission pattern from a mono-peak type to a broad type in accordance with temperature increment. The RTL pattern heated at 800°C agreed well with the pattern of the Hakkoda ignimbrite, presumably showing the emplacement temperature of 800°C in the volcanic products.

Keywords: Red thermoluminescence dating, Pleistocene tephra, RTL emission pattern

## The comparison of Red Thermoluminescence dating and Radiocarbon dating

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A widespread tephra layer is often used as a "time marker bed" in geology and geography. They can be dated by radiocarbon method on organic materials to determine the timing of volcanic events occurred up to ca.50 ka. However, lacking of suitable material (e.g. burial wood) for radiocarbon dating is often become an obstacle to directly estimate the timing. Alternative option is using the K-Ar method, yet considerable uncertainties for dating of younger samples (< 100 ka) and limit of the dating materials, namely the requirements of high K contents minerals, also prevent from the methods ubiquitously be applicable for tephra dating. Although the period between 50 to 100 ka is very important for various studies such as Paleoclimatology and Archaeology, the chronological "gap" has been existed.

A trapped radiation charge dating technique will potentially solve these problems. We focused on development of the Red Thermoluminescence (RTL) dating for tephra since we observed that it captures stable and high intensity RTL signal. We established the dating protocol to deal with tephra to conduct RTL dating. Comparing with ages of tephra dated by radiocarbon and K-Ar agreed very well with RTL dates showing reliability of our newly developed experimental protocol. Uncertainties of the RTL ages were much reduced and they were comparable to the radiometric dating results.

Keywords: AT ash, radiocarbon dating, Red thermoluminescence dating, widespread tephra



## Characteristics of various feldspar IRSL signals and their applications

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Recent studies have revealed various recombination processes of feldspar infrared stimulated luminescence (IRSL) signals measured at different preheat and stimulation conditions. These different IRSL signals have very diverse thermal, athermal and bleaching properties. The post-IR IRSL (pIRIR) signals measured at 150°C (pIRIR<sub>150</sub>), 225°C (pIRIR<sub>225</sub>) and 290°C (pIRIR<sub>290</sub>) are most commonly used for dating sediments. However, no widely accepted criteria exist to select preheat and stimulation temperatures for samples with different age ranges and from different depositional environments. It has been known that the pIRIR signal with higher stimulation temperatures is more thermally and athermally stable than the lower temperature signal, however, the higher temperature pIRIR signal is much more difficult to bleach. Therefore the higher temperature pIRIR signal (e.g. pIRIR<sub>290</sub>) is less suitable for dating sediments from difficult-to-bleach environments. The pulsed IRSL signal is also known to be more stable than the conventional IRSL signal. Since the pulsed measurement is performed at a low stimulation temperature, this signal appears to be much better bleachable than the pIRIR signal and therefore more suitable to date waterlain sediments. In the presentation I will also introduce newly developed OSL thermochronology using multiple IRSL signals stimulated at different temperatures, which have different thermal stabilities.

Keywords: luminescence dating, feldspar, OSL thermochronology

## Thermoluminescence dating of calcite: Application to calcite vein deposited from groundwater in Luzon, Philippines

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Advantages of calcite thermoluminescence dating are (1) effective range of dating is from thousands to one million, suitable to quaternary research, and (2) the radioelement in sample is not necessary, therefore applicable to variety of materials. Thus, thermoluminescence dating can be applied to calcite as well as  $^{14}\text{C}$  dating and/or uranium disequilibrium. TL dating has been applied to various calcites, for example, inorganic carbonates such as limestone and stalagmite, and biogenic calcite such as shell, coral and snail plate. However, thermoluminescence dating of calcite is sometimes problematic; e.g., sensitivity change occurred through repeated heating of samples, difference in characteristics of luminescence response against different kinds of radiation (e.g., alpha-ray, beta-ray, gamma-ray and X-ray). This study applied TL dating to calcite vein deposited from high alkaline groundwater originated probably from the serpentinization of mafic rocks in Luzon, Philippines. High alkaline groundwater has been circulating along cracks in a rock and bentonite layers. Age of calcite contribute to determine the timescale of fluid-bentonite interaction in a geological framework, which is an important knowledge for a deep geological repository of radioactive wastes, for bentonite plays an important role to prevent an outflow of the contaminated groundwater to the environment.

For paleodose measurement, SARA (single-aliquot regeneration and added dose) method was applied to evaluate sensitivity change of calcite occurred through repeating heating of samples. To know the difference in characteristics of luminescence response against different kinds of radiation, we measured thermoluminescence of sample induced by alpha-ray, beta-ray, gamma-ray and X-ray, and the ages of calcites were calculated with these results.

This study is a part of a project to develop an integrated natural analogue programme in the 2011-2014 fiscal years in Japan, which was funded by Agency for Natural Resources and Energy of the Ministry of Economy, Trade and Industry, Japan.

Keywords: Thermoluminescence, Calcite

## Examination of dose dependence for quartz-TL using automated reader with four optical paths

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From minerals such as quartz, thermoluminescence (TL) is emitted from electrons in the electron traps as they recombine with holes in the recombination center. When a mineral crystal has recombination centers of several types, TL of several wavelengths with different cross-sections might be emitted. Consequently, the dose-dependence of a particular TL might differ from other TL of a different wavelength. When applying TL to dating, linear dose dependence is preferred along with strong TL sensitivity (Nagatomo *et al.*, 1999).

We designed and constructed a TL/OSL (NUE-05-OSLTL) reader that has four light paths with four condensing lenses and four PMTs for choosing the suitable luminescence wave range for dating (Shitaoka, 2015). The wave ranges of the four lens and optical filter assemblages are usually 300-390 nm, 390-590 nm, 570-700 nm, and 350-700 nm.

This study used an NUE-05-OSLTL reader to measure mafic lavas in the Oninomi monogenetic volcano in northern Kyushu, Japan, which include small amounts of quartz xenocrysts (Ohta *et al.*, 1992).

The TL-dose dependences from four wave ranges will be presented during the poster session.

Keywords: Thermoluminescence, dose dependence, linearity, quartz, Oninomi lava

## Measurement of thermoluminescence efficiencies induced by alpha, beta, gamma and X-ray using synthetic calcite

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In comparison to quartz thermoluminescence, characteristics of calcite thermoluminescence is less understood, thus, thermoluminescence dating was not often applied to calcite in recent years. Earlier studies suggested that characteristics of calcite thermoluminescence depend on impurity concentrations, however, it is not quantitatively understood and the difference in characteristics of luminescence response against different kinds of radiation is not clear.

By analyzing chemical composition and luminescence efficiencies induced by alpha, beta, gamma and X-ray of natural calcites, it is suggested that luminescence efficiency factors are a function of Mg, Mn and Fe concentrations. In this study, synthetic calcites with controlled impurity concentrations were analyzed to evaluate relationship between multiple impurity concentration and thermoluminescence efficiencies by each radiation (alpha-ray, beta-ray, gamma-ray and X-ray) quantitatively.

In Kanazawa University, X-ray is used as artificial radiation source to prepare calibration curve. A known dose was given by each radiation source (alpha-ray; <sup>241</sup>Am, beta-ray; <sup>90</sup>Sr and gamma-ray; <sup>60</sup>Co) and then estimated by the SAR method using X-ray. The luminescence efficiencies by each radiation normalized to X-ray (a-x-value, b-x-value and c-x-value) were calculated with given dose and measured dose.

The c-x-values of Mn doped calcites are lower than that of non-doped and Mg doped calcites, and the c-x-values are negatively related with Mn concentration. The c-x-value of Mn-Mg and Mn-Fe doped calcites are slightly lower than that of Mn doped calcite, however, are independent of Mg or Fe concentration. As a result, Mn works to decrease c-x-value, and contributions of Mg and Fe to c-x-value are negligibly small.

Results of a-x-value and b-x-value also will be presented on our poster.

Keywords: Thermoluminescence, Calcite

## Characteristics of ESR and TL signals of quartz in the present river bed sediments and in possible source rocks around Kizu River

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ESR, TL and OSL signals have been used for the dating of samples in Quaternary [1], [2], [3]. Recently another direction has been tried, using the ESR and TL signals as indicators of sediment provenance. The ESR signal intensity of quartz is shown to be a useful parameter to investigate the provenance of aeolian dust [4], [5]. The ESR signal intensities of quartz have been shown to be useful to distinguish the sediment provenance [6]. Quartz has been reported to show red and blue TL by the differences in origin [7].

Sediment provenance gives important information on the erosion processes, river contention, and crustal movement and so on, suggesting the environments at the time of sediment transportation. By examining the quartz crystals found in sediment and related bedrock, it may be possible to estimate the provenance of sediment.

In this study, we report the characteristics of ESR / TL signals of quartz in the present river bed sediments and in the possible source rocks, to discuss the possibilities of identifying sediment provenance.

All quartz samples were irradiated by gamma ray to a dose of 2.5kGy. ESR signals were observed by ESR spectrometers (JES-X320; X-band JEOL RESONANCE Inc.). TL signals were observed by selecting the wavelength region by using the Time-Resolving Spectroscopy System.

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Keywords: thermoluminescence, Electron Spin Resonance, sediments provenance, quartz, river bed sediments

## OSL dating and tephra analysis of fluvial terrace sediments

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Estimating the geological environments associated with uplift and erosion is important for the safety assessment of the geological disposal of high-level radioactive waste in Japan. The uplift rate during the past hundred thousand years is estimated from the elevation and emergence ages of terrace surfaces which are used as geomorphologic standard. Optically stimulated luminescence (OSL) dating is effective method to determine the emergence ages because it can be applied to common mineral grains (quartz and feldspar) present in sediments. In recent years, OSL dating is generally used in the continental regions such as Europe and Australia, but the application in Japan is far behind. In particular, the application of OSL dating to fluvial sediments is still limited in Japan. We therefore report a case study of quartz OSL dating of fluvial terrace sediments, and its adequacy is considered in the correlation with intercalated marker tephra. All samples were collected from fluvial terrace sediments along Kiso River and Tsukechi River which is a tributary of the Kiso. The investigated fluvial terrace sediments are composed of laminated sand layer and pumice layer which is likely to be reworked. The pumice was identified as On-Pm1 tephra (100ka; Kobayashi et al., 1967; Takemoto et al., 1987) by mineral composition, refractive indices of volcanic glass and phenocryst minerals, and the major element composition of volcanic glass. This indicated that the fluvial terrace sediments were deposited after 100ka. The extracted quartz grains from terrace sediments were measured by conventional SAR protocol (Murray and Wintle, 2000). The obtained OSL ages are about 40~50 ka, as far as we can foresee, being underestimated because the OSL decay curves from all samples were dominant by medium component which is unsuitable for OSL dating. We thus address to isolate a fast-component which is suitable for OSL dating, and the equivalent dose derived from the fast-component OSL is discussed in the presentation. This study was conducted under a contract with METI (Ministry of Economy, Trade and Industry) as part of its R&D supporting program for developing geological disposal technology.

Keywords: OSL dating, Fluvial terrace sediments, On-Pm1

## Luminescence dating of lacustrine lowland sediments around Lake Kasumigaura

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Lake Kasumigaura is a coastal lake in southeast Ibaraki Prefecture, and has lacustrine lowland formed in response to sea-level changes around its lake shoreline. While several studies have been conducted to explore the formative processes of the lowland which is critical for understanding of the paleoenvironment, little is known about absolute age of the lowland sediment. In this study, we applied luminescence dating to the lowland sediments of Lake Kasumigaura.

Nine sediment cores were obtained using a 2-m-long geoslicer in lacustrine lowland at Gochoda, Namegata. Six sediment samples were then taken from selected cores for the luminescence dating and processed in subdued red light. The samples were sieved and treated with chemicals to extract the 120-250  $\mu\text{m}$  quartz and K-feldspar fraction. In preliminary measurement, we found that the quartz OSL signal is dominated by component unsuitable for dating, and thus decided to carry out K-feldspar IRSL and post-IR IRSL (pIRIR) protocols for equivalent dose ( $D_e$ ) estimation. As a result of several test, the preheat and cut heat temperature of 200°C, hot IR breach of 205°C were suitable to measurements. As the lowland sediment is expected to have Holocene age according to previous studies, we used a relatively low pIRIR stimulation temperature, 170°C, which is known to be suitable for young samples. The bleaching and fading tests of pIRIR showed that the residual dose was about 1 Gy, and that the g-value was about 0-1 %/decade, respectively. Thus, for the age estimate of pIRIR, the residual dose should be subtracted from the  $D_e$  estimate while no fading correction is required. The IRSL in contrast showed considerable anomalous fading; its g-value was around 10%.

It was revealed the both of pIRIR and IRSL result that  $D_e$  tend to be higher with increasing depth at a single core site. The largest  $D_e$  of pIRIR was about 50 Gy from the deepest sample of the core nearby the Pleistocene terrace. Other samples showed  $D_e$  s ranging from 1 to 15 Gy. We will consider radionuclide concentration quantified by inductively coupled plasma mass spectrometry for the determination of the environmental dose rate and then calculate the pIRIR and IRSL ages. We will also compare the age estimate with <sup>14</sup>C age.

Keywords: OSL dating, pIRIR dating, Kasumigaura, lacustrine lowland