Quartz OSL dating of deep marine sediment: how accurate and how far back?

A case study from the Sea of Japan

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Quaternary hemi-pelagic sediments in the Japan Sea record millennial and orbital scale changes in the East Asian Monsoon as alternations of dark and light layers. To explore the development and evolution of monsoonal patterns and their relationship to past changes in the Japan Sea, cores were drilled at Sites U1424 and U1425 during the IODP Expedition 346. Based on the data from Site U1424, a sediment age model was constructed using tephro-stratigraphy, and magneto-stratigraphy and the sediment colour profile tuned to the d^{18}O profiles of both LR04 and Chinese stalagmite (Lisieki and Raymo, 2005, Cheng et al., 2016). The tuned age model for Site U1424 over the last 640 ka is consistent with that from LR04 within its age uncertainty of <5 ka (Tada et al., 2017).

In this study, we have used OSL dating of fine-grained detrital quartz (of aeolian origin) to provide high-resolution numerical age models for the late Quaternary in the cores from Sites U1424 (hole U1424C) and U1425 (hole U1425C). The objectives of this study are 1) test the quartz fine-grained OSL dating limitation, 2) test the reliability of the OSL ages by comparison with tephro-stratigraphy and the orbitally-tuned age model.

The dose recovery ratio is 0.95±0.01 (n=178) for samples from core U1425C and 0.913±0.01 (n=248) for U1424C for a 180˚C/10s preheat and140˚C cut-heat. Dose rates were determined using high resolution gamma spectrometry, and vary considerably, from 1.04 to 4.19 Gy/ka. The equivalent dose from both cores increases with depth, up to 700 Gy. However, the OSL ages appear to underestimate from ˜150 ka, with saturation at ˜250-300 ka. This underestimation may be related to systematic underestimation of field water content (and so overestimation of dose rate) at depth. The ages indicate a more or less constant sedimentation rate back to ˜120 ka for U1424C. This is in contrast to core U1425C which shows a pronounced change in sedimentation rate at ˜120 ka and ˜40 ka for U1425C. The OSL ages are in good agreement with Toya, Aso-4, Aira-Tn tephra at 112, 87 and 30 ka, and with the orbitally-tuned age model, for the U1424C. However there are discrepancies during MIS 4/3 and 2/1 in U1425C, where the OSL ages suggest increased sedimentation rates; these are not present in the orbitally-tuned age model. Given the good agreement between the two approaches in U1424C, where the sedimentation rate appears to be relatively constant, there seems no reason to doubt the reliability of the more direct OSL data in U1425C.

Keywords: optical dating, quartz fine grain, Japan Sea

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Luminescence dating of marine terrace deposits in Noto Peninsula and inferred uplift rate

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Marine terraces formed in relations to sea-level highstand are important for understanding tectonics in the coastal area. Their chronology, along with paleo sea-level indicator, plays a critical role in estimating the uplift rate. Marine terraces have been correlated to Marine Isotope Stage (MIS) 5c, 5e, 7 and 9 in the Noto Peninsula, but no comprehensive absolute dating has been practiced except for U/Th dating of coral materials in the northern part. We carried out the pIRIR$_{200/290}$ dating of K-rich feldspar from marine deposits comprising the marine terraces to check the correlation. The depositional succession of the MIS 5c terrace in the northern part consists of the lower muddy incised-valley unit and upper sandy shallow-marine unit, the boundary of which is characterized by an erosional surface. The pIRIR ages of the lower and upper units are 137±7ka and 102±3ka, respectively. Considering the error range, the lower and upper units are thought to have been formed in MIS5e and MIS5c, respectively. This is concordant with the U/Th age of the lower part and tephrochronology. These ages further indicate a significant hiatus across the erosional boundary. No distinct marker of paleo sea level is observed in the succession, and only the minimum uplift rate is estimated as 0.41 m/ky from the level of marine deposit and highstand sea level in MIS 5c. Ocean current despots are identified below marine terraces correlated to MIS 7 and 9 in the southeastern part of the peninsula, and their pIRIR ages are 220±18 ka and 317±27 ka, being concordant to the terrace correlation, respectively. These suggest that there used to be a strait between the Noto Peninsula and Honshu Island at least during the interglacial sea-level highstand before MIS 7, allowing the northeastward ocean current. The strait was then closed due to the subsequent uplift. No distinct sea-level marker is not obtained in these terrace deposits, and the minimum uplift rates are estimated as 0.17 m/ky after MIS 7. Another marine depositional succession below the terrace correlated to MIS 9 is dated 293±21 ka, appearing to be slightly underestimated than MIS 9. Assuming that the terrace was formed during MIS 9, the minimum uplift rate is 0.13 m/ky. Our pIRIR ages of marine terrace of MIS5c, 7 and 9 are concordant with the previous correlation, and suggest the higher uplift rate in the northern part of the Noto Peninsula.

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Keywords: Luminescence dating, Marine terrace, Noto Peninsula, Uplift rate

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Characteristics of luminescence signals from insoluble residue in raised coral reefs

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Dating of aragonitic coral is important in studies of sea level and tectonic history in the tropical and subtropical coastal area. Uranium-series (U-series) and Electron Spin Resonance (ESR) dates of coral have been providing us the detailed Quaternary sea level history [1]. Aragonite of coral fossils, however, often alters into calcite during the period of burial, interfering with 14C, U-series and ESR dating. In this study we dissolved altered corals by hydrochloric acid and investigate characteristics of luminescence signals from the residual materials to discuss possibilities for an elevated-temperature infrared stimulated luminescence (post-IR IRSL) dating. If we can estimate the age of coral from the insoluble residue, it will be of great help not only for cross checking the U-series or/and ^{14}C age but for estimating the age of altered coral fossils which can not be dated by them.

The fossil coral samples were taken from Yonaguni Island, southwestern Ryukyus, southwestern Japan. The sampling layer is considered as middle –late Pleistocene horizon [2]. In order to get materials from the limestone for blue light stimulated luminescence (BLSL) or post-IR IRSL dating, the outer-rim of the sample, which might be bleached by sunlight, was cut off more than 5 mm under subdued red light in a darkroom. Furthermore the sample was dissolved in 6N Hall in 10 minutes to remove the outer surface because some parts of the sample surface were rugged and difficult to cut off completely. Our preliminary experiment suggested the acid treatment of 6N HCl in 10 minutes removed more than 5mm depth from the sample surface, we think this procedure made sure to extract unbleached materials from the sample. Then the sample were washed by distilled water and again soaked in 6N HCl with hydrogen peroxide (several %) till the calcite was completely dissolved. After these treatments above, we could get small amount of residual materials from the coral fossil sample.

In this presentation we show some characteristics of luminescence signals from these residual materials and discuss possibilities for luminescence dating[3],[4].

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Quartz OSL properties from coastal sand in Eastern Japan

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Optically stimulated luminescence (OSL) has been widely applied to quartz in sediments. However, quartz that has unstable OSL signal components can be found in tectonically active regions such as Japanese islands (e.g. Tsukamoto et al., 2003; Tokuyasu et al., 2010; Tamura et al., 2015) and it can cause problems for OSL dating. Thus, it is needed to consider the OSL properties carefully if quartz with such properties is measured. Quartz origin is one of possible factors contributing to the OSL properties (e.g. Tokuyasu et al., 2010), but relationship between the quartz origin and OSL properties is not discussed well enough. We therefore investigated OSL properties of coastal sand from Tohoku to Kanto region. As a result, the characteristics of OSL signal from adjacent sampling locations indicate similar trend. Although geomorphological features should be considered, it is likely that the OSL properties of coastal sand roughly reflect the sediment provenance and that the properties come from the average OSL signal for the original rocks.

Keywords: quartz OSL property, sediment provenance

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Importance of quartz thermoluminescence peak temperature

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The peak temperature and the intensity of the emission signal is necessary for calculation of the dose by the thermo-luminescence (TL) method using quartz.

The shape of the luminescence signal becomes broad when a sample has several luminescence sites, and the apparent peak formed by several luminescence sites with different stability may not be useful for dating of samples with complex thermal history.

In such a case, it is necessary to separate peaks and calculate accumulated dose by single luminescence site. For peak separation, the original peak temperature must be necessary. The peak temperature is estimated by the T-Tmax method (McKeever, 1980). In this method, the peak temperature is observed by erasing the signal below pre heat temperature. Generally, as the heating rate increases, the peak temperature shifts to a higher temperature (Aitken, 1985).

The peak temperature is different depending on the sample. In general, peak temperature is the same for the natural and the regenerative signals. However, they are different in some samples.

In this research, we will discuss the difference in peak temperatures of the natural signal and the regenerative signals.

Keywords: Luminescence, peak temperature, quartz

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Characteristics of calcite thermoluminescence: Radiation and luminescence efficiency

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Thermoluminescence dating method as well as ^{14}C and U methods has been applied to calcite, but it is less popular partly because the difference in luminescence response for different kinds of radiation is not clear. To report more reliable thermoluminescence ages from for calcite, fundamental characteristics of its response to radiation exposure were investigated and related to chemical composition by analysing natural and systhetic calcites with controled impurity concentrations. Relative thermoluminescence efficiencies for calcite by beta or gamma irradiations for calcite against quartz are under 1.0, and it indicates that equivalent dose of calcites samples were underestimated when calibration curve was created with X-ray source calibrated using quartz. This may be caused by differences in common substitution elements in calcite versus quartz. Interaction between mediums and radiation is affected by radiation energies more sensitively for calcite than quartz. [NH1] Gamma efficiency is a function of total Mn, Mg, and Fe concentrations. Thermoluminescence efficiency by beta ray must be considered together with the cathodoluminescence emission. Fe, which works as quencher in cathodoluminescence, may keep prevent release of beta radiation energy from beta ray instead of transmitting to activatoras a form of cathodoluminesence and increase the thermoluminescence efficiency. The estimate of accumulated dose from alpha rays is affected by sample thickness because of the spatial energy density around the center of the alpha track and the luminescence detection range. Thus, for accurate alpha efficiency measurements, evaluation of the effective alpha ray range and luminescence detection thickness is important. The k-value (alpha efficiency against absorbed gamma ray dose rate) increases with Mn concentrations.

Keywords: calcite, thermoluminescence efficiency

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ESR and radioactive disequilibrium dating of sulfate minerals in sea-floor hydrothermal deposits at the Okinawa Trough

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The time scale how long the sea-floor hydrothermal activities continue is an important issue in the studies on the processes of ore formation and on the evolution of the biological systems sustained by the chemical species arising from hydrothermal activities. For this purpose, radioactive disequilibrium dating methods such as U-Th for sulfide minerals (e.g. You and Bickle, 1998), ^{226}Ra-^{210}Pb and ^{228}Ra-^{228}Th methods for barite (e.g. Grasty et al., 1988, Noguchi et al., 2011) have been used, while recently, the ESR (electron spin resonance) method for barite was proposed and developed (Okumura et al., 2010, Fujiwara et al., 2015).In the present study, we applied ESR and radioactive disequilibrium dating of sulfate minerals, i.e., anhydrite and barite.

Firstly, for anhydrite ($CaSO_4$), we will report the first successful dating results on hydrothermal anhydrite using the ^{228}Ra-^{228}Th method. The hydrothermal ore samples were taken by research cruises operated by JAMSTEC. The anhydrite crystals were physically scratched out of the samples. The radium (^{226}Ra, ^{228}Ra) were measured in the same samples for the ESR method by the low background gamma ray spectrometry. From the activity ratios, disequilibrium ages were obtained to be 0.6-2.5 years by ^{228}Ra-^{228}Th method. As anhydrite is unstable because of the higher solubility increase at low temperature, obtained ages of the anhydrite samples are reasonable.

Secondly, for the barite ($BaSO_4$), three dating methods of ESR, ^{226}Ra-^{210}Pb and ^{228}Ra-^{228}Th methods were applied to the same hydrothermal barite samples. The ESR ages of barite taken from hydrothermal areas in the Okinawa trough range from 4.1 to 16000 years, filling the age gap of the maximum age, 150 years, of ^{226}Ra-^{210}Pb method and the minimum age, several thousand years of U-Th method, being the most appropriate age range to discuss the evolution of the hydrothermal systems. Interestingly, the ^{226}Ra-^{210}Pb and ^{228}Ra-^{228}Th ages for the same samples are the same or younger than the ESR ages. The difference in the ESR and two radioactive disequilibrium ages would most probably due to two or more formation stages of the barite crystals in the sulfide deposits (Uchida et al., 2015). This interpretation is supported by the BSE images where at least two kinds of the barite crystals of different occurrence are observed. Model calculations including two or more formation stages (possibly continuous) may consistently explain the differences among the ESR and the disequilibrium ages, revealing the histories of the episodic hydrothermal activities.

Keywords: barite, anhydrite, hydrothermal activities, electron spin resonance, dating

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Estimation of the mixing ratios of quartz in the possible source rocks that make up the present river bed sediments by using ESR

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Bedrock is broken down by weathering into unconsolidated materials, which are then carried downstream by rivers. When a new procedure for clarifying provenance of sediment is established, it can give significant information on the erosion processes, river contention, and crustal movement suggesting the environments at the time of sediment transportation.

Recently, Electron Spin Resonance (ESR) signals are used as indicators of sediment provenance. The E_1’ center signal intensity of quartz is shown to be a useful parameter to investigate the provenance of aeolian dust [1] - [3]. The Al, Ti–Li, and E_1' center signal intensities of quartz are useful in determining sediment provenance [4]. The Al and Ti–Li center signal intensities of quartz are possible to estimate the mixing ratios of sediment provenance [5].

In this study, we will report estimation of the mixing ratios of the source materials that make up the present river bed sediments by using ESR signal intensities of Al, Ti-Li and E_1’ centers.

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Keywords: ESR, Quartz, Provenance of sediments, Mixing ratios of sediments, Present river bed sediments

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Cathodoluminescence and electron spin resonance characterization of synthetic quartz

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Cathodoluminescence (CL) and electron spin resonance (ESR) detect structural defects and impurities in quartz. The E_1’ center can be easily identified by ESR method. In the ESR, the E_1’ center is defined as an unpaired electron in a single silicon sp^3 orbital oriented along a bond direction into an oxygen vacancy (Feigl *et al.*, 1974). However, the assignment of E_1’ center in CL has been discussed so far in comparison with the results by ESR method (*e.g.*, Stevens-Kalceff, 2009). Usually, natural quartz has various impurity elements and defects. In this study, we have conducted to clarify the E_1’ center in synthetic quartz by comparison with CL and ESR.

Single crystal of synthetic quartz without any impurities (Miyazaki Epson Co.) was employed for CL and ESR measurements. Sample was sliced perpendicularly to the c axis, and its surface was polished. Electron were irradiated on the surface using a Cockcroft-Walton accelerator located at Takasaki Research Center (JAERI). Implantation energy of electron beam was at 1.0 MeV at 5 stages of 4.51×10^3 -3.20×10^4 J/cm^2. CL color images were obtained using the Luminoscope (ELM-3). CL spectroscopy was carried out by an SEM-CL system, which consists of an SEM (JEOL, JSM-5410LV) combined with a grating monochromater (Oxford, Mono CL2). All CL spectra were corrected for total instrumental response, which was measured using of a calibrated standard lamp. ESR spectra were obtained by an ESR spectroscopy (JEOL, JES-PX2300).

The ESR spectra obtained from annealing sample at 600 ºC exhibit no signal assigned to E_1’ center at g = 2.001, whereas weak one is recognized in untreated sample. According to Toyoda *et al.*, (1996), the heating up to 600 ºC almost quenches the signal of E_1’ center. The results of the ESR measurements indicate that the signal of E_1’ center in both annealed and untreated samples pronounces with an increase in the irradiation dose. It clearly shows a formation of E_1’ center. in quartz by electron irradiation. CL analysis by a spectral deconvolution method reveals five emission components related to defect centers at 1.77 eV, 1.9 eV, 1.95 eV, 2.3 eV, 2.7 eV and 3.14 eV for annealed and untreated samples. An increase in electron-irradiation dose results in an increase in the intensity of the component at 1.77 eV. This result suggests that the emission at 1.77 eV in a red region closely relates to the E_1’ center determined by ESR method, whereas the emission component at around 2.7 eV has been assigned to the E_1’ center involved with STE in quartz.

Keywords: quartz, cathodoluminescence, electron spin resonance, E1' center

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Evaluation of relationship between fault displacement and ESR intensity using low-speed ring shear apparatus

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It is important to evaluate fault activity for mitigating risks of the disasters. In this study, we focus on fault dating by electron spin resonance (ESR) method. This method is explained easily in the following steps. First, number of electrons and halls trapped in defects in quartz are detected as ESR signals. Second, a total dose is calculated from the intensity of ESR signals. Third, the total dose is divided by an annual dose rate to obtain elapsed time from the latest earthquake on the fault, because the ESR intensity is reset to zero under a temperature of about 300 or 500 degrees to which the fault is heated by coseismic slip. Therefore, ESR dating is one of the appropriate techniques to assess fault activities. However, the hypothesis that numbers of electrons and halls trapped in defects are reset to zero by heating has not been tested thoroughly. Moreover, it is not clear whether zeroing of the ESR intensity can happen on shallow faults from which we can easily obtain samples, because frictional heat and terrestrial heat are negligible on such shallow faults.

There exist several studies on a relationship between fault displacement and ESR Intensity (Tanaka, 1987; Hataya and Tanaka, 1993; Fukuchi, 2004). However, they have the following issues: (1) Conditions of ESR measurement were inappropriate. (2) Specific ESR signal was only measured and other signals were not. (3) Relationship between displacement and ESR intensity was not clearly represented by either a table or a plot. (4) Effect of heat on ESR intensity was not considered. Therefore, we conducted shear tests with silica sand (JIS test powder) under the conditions of shallow depths where frictional heat is negligible to clarify a relationship between displacement and ESR intensity. The inner diameter and outer diameter and thickness of sample are 20 mm, 30 mm and 1.5 mm. We used low-speed ring shear apparatus in Tohoku University. Sliding rate and the normal stress were kept constant to 0.72 mm/s and 0.981 MPa, respectively. Final displacements were varied among test runs up to 1.7 m.

Temperature rise δT on the shear surface was estimated to be a few degrees from Sibson (1975) under the conditions of this experiment. So, an effect of heating is negligible in this study. ESR measurement was conducted under appropriate conditions in which E_1' center representing ≡Si (means a lone electron and - means sheared electron pair, and ≡means one Si atom combined with three Si atoms by each electron pair) does not overlap with R signal (structure corresponding to this signal is not clarified yet) and the other ESR signals can be detected. The relationship between displacement and ESR intensity was investigated by ESR measurements before and after the shear displacement. Many ESR signals such as E_1' center, OHC (O^{3-}), Peroxy center (≡Si-O-O) and so on decreased as displacement increases. This means that accumulation of displacement contributes to zeroing of ESR intensity under a low confining pressure corresponding to a sub-surface. Therefore, it is possible to reset the ESR intensity by fault activities at shallow depths.

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