

Luminescence dating –what is it, what can it do, and why is it important?

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The Earth's surface is an archive of the history of our species and of our environment but to read and interpret the information recorded in this archive, we must have a means of knowing when the various records were preserved, and so when the events actually happened. Luminescence dating is an important and widely-applicable chronological tool used to date these records. The technique is not new, but recent developments have led to an explosion in applications, so that today it is one of the three most widely used methods in the geo- and archaeo-chronology of the last 500,000 years. This talk outlines the principles of the method, and discusses the advantages and limitations of the most widely-used form, optically stimulated luminescence (OSL) dating. Evidence for the precision and accuracy of the method is presented. The importance of the technique to studies of human evolution and migration, and to our understanding of past climate change is then illustrated using studies ranging in scale and time from the recent bioturbation of mudflats to late Quaternary ice advances in Eurasia. Finally, exciting new developments in rock surface dating are summarised.

It is concluded that OSL dating in its various forms is the most widely applicable dating tool available to earth scientists and archaeologists. It has grown from being relatively minor and unimportant to become one of the three pillars supporting modern archeo- and geo-chronology, and despite nearly 60 years of development, new signals, new techniques and new applications are constantly appearing. It continues to be a very exciting field in which to work.

Keywords: OSL dating, geochronology, quartz feldspar, rock surface dating

海浜カットアンドフィル堆積物の高分解能OSL年代測定：オーストラリア南西部Bengello Beachにおける海浜侵食履歴の評価

High-resolution OSL dating of cut-and-fill beach deposits for assessing beach erosion history in Bengello Beach at Moruya, SE Australia

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海浜や砂丘地などの露光しやすい環境では、石英の光ルミネッセンス (OSL) により、過去数十年程度の若い堆積物の年代を決定することができる。ごく稀に大規模なストームが発生すると海浜の砂が侵食されて海岸線が大きく陸側後退することがあり、そうした侵食の規模や頻度を理解することは海岸の保全に重要である。しかし、現在の海浜で行われている観測の期間は短く、そうした低頻度のイベントを経験的にとらえることは容易ではない。海浜堆積物は、過去の長期間に渡る堆積と侵食との繰り返しにより形成されるものであるが、それをもし連続的に年代測定することができれば、そこから海浜侵食の履歴を読み取ることができる可能性がある。ここでは、ベイズモデルを組み合わせた高分解能光ルミネッセンス年代測定と地中レーダ探査に基づく地質学的な海浜侵食履歴の評価方法を、オーストラリア南西部のBengello Beachを例に示す。Bengello Beachでは1972年以来定期的に海浜地形の観測が行われており、大規模なストームの後に海浜が後退しその後2~3年以内にまた回復するという、カットアンドフィルと呼ばれる地形変化パターンを示すことが知られている。1974年に発生したストームにより海浜が50 mも後退したが、その後は海浜の後退量が30 mを超えるストームは発生していない。このため、1974年の海浜侵食は50~100年程度の再来周期を持つ低頻度のイベントであると考えられている。この海浜地形の観測は、海浜堆積物の上部の中で海浜侵食の直後の回復の期間に形成されたもののみが地層として保存されることを示している。つまり、海浜堆積物上部の年代は、過去に起こった大規模な海浜侵食のタイミングをほぼ表しているとみなすことができる。OSL年代の試料は、Bengello Beachの前置砂丘から内陸部120 mまでに発達する浜堤の地下から5~10 mの水平間隔で採取した。ここで最も陸側の古い試料は510年前の年代を示すため、それ以降の正味の海岸線の前進速度は0.24 m/yとなり、中期完新世以降の平均値に等しい。得られたOSL年代は、350, 180, 130, 90年前の侵食イベントを示唆し、また1974年の侵食により形成された地形と整合的な結果となった。海浜の前進速度が0.24 m/yで一定だと仮定すると、これら4回のイベントでの後退量は45~55 mと見積もられ、1974年に匹敵する。350年前のイベントに続いて形成された幅約40 mの区間の海浜堆積物から採取された4つの試料はほぼ同じ年代を示し、そのすぐ海側の堆積物との間で150年間のギャップが認められ、180~330年前においては大規模な海浜侵食が発生しなかったことを示唆している。以上の高分解能OSL年代測定により、Bengello Beachで1974年に匹敵する海浜侵食は、350年前以降50~150年の間隔で発生していることが明らかになった。

キーワード：海岸、年代学、堆積学、第四紀学、ルミネッセンス年代

Keywords: coast, chronology, sedimentology, Quaternary science, luminescence dating

Luminescence characteristics and IRSL-chronology of extreme-wave event deposits recorded at the Shirasuka lowlands, Japan

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The location of Japan at the Pacific-Philippine-Eurasian subduction zones makes it vulnerable to earthquakes and subsequent tsunamis. Furthermore tropical cyclones cause destructive storm surges. Both natural hazards may generate extreme-wave events, which are a major threat for coastal communities.

The Shirasuka lowlands, sandwiched between a Mid-Pleistocene terrace and a coastal dune, record evidence for numerous extreme-wave events. Located along an important historical trade route, their impact history is well documented in written sources and a radiocarbon chronology has been developed for seven extreme-wave event deposits previously identified in this area (Fujiwara et al., 2006; Komatsubara et al., 2008). Therefore, this study area provides an excellent opportunity for testing the applicability of OSL dating to young (< 800 years) coastal, potentially incompletely bleached extreme-wave event deposits.

Quartz is preferred for dating such sediments, due to its faster rate of signal resetting. However, OSL measurements failed due to low signal intensities, absence of a fast component, and sensitivity to IR stimulation. Consequently, feldspar was used instead. The IRSL₅₀ signal has high signal intensities and resets quickly. However, thermal transfer affects these young feldspars. To minimise this effect and thus reduce recuperation, a second optical stimulation at 130 °C was included in Lx and Tx cycles of the IRSL₅₀ protocol.

Final dating was performed on single-grains of feldspars to (i) lower residuals, and (ii) account for potentially incomplete bleaching.

The resulting ages cover the known historical record of the extreme-wave events of the last 800 years at Shirasuka. Sand sheets can be correlated with tsunamis in AD 1361, 1498, 1605 and 1707. A poorly bleached equivalent dose distribution of the uppermost sand sheet hints at a different transport mechanism. The IRSL age range suggests a correlation with the Tonankai earthquake in AD 1944. Since the subsequent tsunami did not inundate the study area, a terrace slope failure due to intense shaking, is suggested for this sand sheet.

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Komatsubara, J, Fujiwara, O, Takada, K, Sawai, Y, Aung, TT, Kamataki, T (2008), *Sedimentology* 55: 1703-1716.

Keywords: feldspar, single grain, tsunami deposits, storm surge deposits

ルミネッセンス年代測定による阿蘇火山のカルデラ決壊洪水発生時期の推定

Constraining the timing of the caldera outburst floods from Aso volcano

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The bouldery volcaniclastic apron sediment (Takuma gravel bed) west of Aso volcano and the overlying tephric loess were studied by Tsukamoto et al (2013) and a preliminary luminescence age constraint of 72-89 ka for the timing of the caldera outburst flood event, which built the volcaniclastic apron, has been reported. In this study we collected additional samples to further constrain the timing of the caldera outburst flood event. Samples were also taken from the Aso-4 ignimbrite, Hotakubo gravel bed consisting of the lower volcaniclastic apron along the Shirakawa River, and the tephric loess deposited above the gravel bed. The luminescence measurements of the tephric loess samples were performed using two post-IR IRSL (pIRIR) protocols with the pIRIR stimulations at 225°C and 290°C. The two pIRIR signals gave consistent ages and the result indicates that the gigantic caldera outburst flood event probably occurred shortly after the Aso-4 eruption, ~86 ka, and the another bouldery Hotakubo gravel bed, which is of cut terrace deposits derived from Takuma gravel bed or of other possible flood event occurred at ~47 ka. The ages will be further compared with the pIRIR ages from the gravel beds themselves. Interestingly, the Aso-4 ignimbrite using the pIRIR signal at 225°C was dated to a much younger age (~40 ka) than the reported eruption age at ~87 ka (Aoki, 2008). This probably indicates that a very long time was needed for the sampled Aso-4 ignimbrite until the temperature reached to the effective closure temperature of the signal (~60-80°C, King et al., 2016).

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キーワード：ルミネッセンス年代測定、post-IR IRSL、テフリックレス、火山性緩傾斜扇状地堆積物

Keywords: luminescence dating, post-IR IRSL, tephric loess, volcaniclastic apron sediment

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 $180^\circ\text{C}/10\text{s}$ preheat and 140°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.

キーワード：ルミネッセンス年代測定、石英微粒子、日本海

Keywords: optical dating, quartz fine grain, Japan Sea

石川県能登半島における海成段丘堆積物のルミネッセンス年代測定と隆起速度評価

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

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海成段丘の海成層は、最近数十万年間の氷期-間氷期サイクルにおける間氷期（高海水準期）に堆積した浅海成堆積物が隆起することでできたと考えられているため、この堆積物の形成時期や標高を調べることで、隆起速度が得られる。能登半島でも過去の高海水準期であるMarine isotope stage (MIS) 5c, 5e, 7, 9などの時期に形成された海成段丘が存在しているが、その年代の根拠はMIS5eにおけるU/Th年代に限られる。そこで、本研究では海成段丘海成層から抽出したカリ長石にpost-IR IRSL (pIRIR) 法を適用し、段丘編年との比較を行い、隆起速度を見積もった。能登半島北部に位置する珠洲市のMIS5cとされる露頭では、侵食面を境にして下位にエスチュアリー～内湾堆積物、上位に浅海堆積物が見られ、それぞれの年代は 137 ± 7 ka, 102 ± 3 kaであった。これらはMIS5eとMIS5cに対比され、先行研究のU/Th年代とも調和的であった。ただし、浅海堆積物中に海面指標は確認されなかったため、上位の海成層のトップの標高に基づき隆起速度の下限値を求め、 0.41 m/kyという結果を得た。能登半島南部に位置する七尾市や志賀町ではMIS7, 9の海成段丘中の海流堆積物から、それぞれ段丘編年とも調和的な 220 ± 18 kaと 317 ± 27 kaという年代を得た。堆積相解析を合わせ、これらの時代は能登半島と本州との間には北東方向の海流の流れる海峡があったが、その後の隆起により陸化したことがわかった。海面指標は得られなかったが、隆起速度の下限値は 0.17 m/kyと 0.14 m/kyであった。また、MIS9のもう一つの露頭では 293 ± 21 kaという少し若い年代が得られたが、これもMIS9と仮定すると、隆起速度の下限値は 0.13 m/kyであった。本研究では、MIS5c, 7, 9の海成段丘でpIRIR年代を求め、それらは従来の段丘編年と調和的であった。また、隆起速度の下限値から推測すると、能登半島では南部より北部の方が隆起速度の速い傾向が見られた。

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キーワード：ルミネッセンス年代測定、海成段丘、能登半島、隆起速度

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

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 ¹⁴C 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 HCl 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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Keywords: raised coral reefs, luminescence signals, insoluble residue

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.

キーワード：石英のOSL信号特性、後背地

Keywords: quartz OSL property, sediment provenance

石英の熱ルミネッセンス発光ピーク温度の重要性

Importance of quartz thermoluminescence peak temperature

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石英を用いた熱ルミネッセンス (TL) 法で蓄積線量の算出を行う際に必要な情報は、発光シグナルのピーク温度と発光シグナルの強度の情報である。

複数のルミネッセンスサイトから発光シグナルがあると、発光曲線の形状はブロードタイプとなり、複数のピークの混合曲線となる。各ルミネッセンスサイトの安定性が異なるため、混合曲線の見かけ上のピーク発光シグナルを用いて年代測定を行った場合、年代を正確に見積もれないという問題が発生することがある。

そこで、ピーク分離を行ってシグナル強度を算出することが必要となる。ピーク分離を行う際には、ルミネッセンスサイトに対応する本来のピーク温度を算出する必要がある。そこで、T-Tmax法を用いてピーク温度の算出を行う (McKeever, 1980)。この手法は、プレヒート (以下PH) によってPH温度以下のシグナルを消去したのちピーク温度を読み取り、その変化からルミネッセンスサイトを認定する方法である。また、一般的に測定時の加熱速度上昇により、ピーク温度も上昇することが知られている (Aitken, 1985)。

このピーク温度は試料によってある程度固定されており、天然発光シグナルと再現発光シグナルとではピーク温度がほぼ同温度である。しかし、本研究で用いた試料の一部において、天然発光シグナルのピーク温度と再現発光シグナルのピーク温度とが異なる試料が見られた。

そこで、本研究では、天然シグナルのピーク温度と再現シグナルとのピーク温度の違いについて議論する。

キーワード：ルミネッセンス、ピーク温度、石英

Keywords: Luminescence, peak temperature, quartz

方解石の熱ルミネッセンス特性研究：放射線種と発光効率 Characteristics of calcite thermoluminescence: Radiation and luminescence efficiency

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Thermoluminescence dating method as well as ¹⁴C 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 synthetic calcites with controlled 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 activator as a form of cathodoluminescence 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.

キーワード：方解石、熱ルミネッセンス発光効率

Keywords: calcite, thermoluminescence efficiency

沖縄トラフ海底熱水域における硫酸塩鉱物のESR及び放射非平衡年代測定 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.

キーワード：重晶石、硬石膏、熱水活動、電子スピン共鳴、年代測定

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

ESRによる現河床堆積物に含まれる起源岩の混合比の推定

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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平野や台地は、地殻・地盤運動や海面変化の影響を受けており、主に河川の土砂の運搬による堆積作用によって形成される。河川は周辺の岩石や堆積物などを巻き込んだ碎屑物を堆積させるため、地形を形成する堆積物の供給起源が複数存在する可能性がある。堆積物の供給起源を推定することができれば、地形の浸食過程、河川争奪や地殻変動など地形学・地質学的過程の解明のための重要な情報を得られることが期待される。

近年、電子スピン共鳴(ESR)法を用いて堆積物の供給起源を検討する研究がなされている。例えば、風成塵中の石英粒子の E_1' 中心信号は、その供給起源を推定する指標として用いられている[1]-[3]。我々はこれまでの研究により、異なる起源の火成岩に含まれる石英粒子の E_1' 中心信号だけでなく、Al、Ti-Li信号も利用することで、それらの特性の違いから、堆積物の供給起源を推定できる可能性があることを示した[4]。さらに同様の手法を発展させることによって、現河床堆積物とその起源と考えられる岩石中に含まれる石英粒子のAl、Ti-Li中心信号を用いて、現河床堆積物の混合比を検討した結果を報告している[5]。本発表では、現河床堆積物とその起源の可能性のある岩石や堆積物中に含まれる石英粒子の複数のESR信号(Al、Ti-Li、 E_1' 中心信号)を利用した現河床堆積物の混合比の推定について報告する。

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キーワード：電子スピン共鳴、石英、堆積物の起源、堆積物の混合比、現河床堆積物

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

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/cm². 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 monochromator (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 °C 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 °C 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.

キーワード：石英、カソードルミネッセンス、電子スピン共鳴、E1' 中心

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

低速リング剪断試験による断層変位量と ESR 信号強度の関係の評価 Evaluation of relationship between fault displacement and ESR intensity using low-speed ring shear apparatus

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地震の被害を最小限に抑えるために重要であるのが断層の活動性評価であり、その1つに電子スピン共鳴 (ESR) を用いた断層年代推定法がある。この方法は石英中の欠陥に捕獲された電子やホールの個数を ESR 信号として検出し、その ESR 信号の強度から求めた総被曝線量を年間線量率で除することで断層最終活動年代を求める方法である。これらの ESR 信号の信号強度は、300 度や 500 度付近での加熱によるゼロセットが起こりうることから、断層運動の評価に有効であると考えられる。しかし、ESR 信号強度が実際の断層運動によって完全にゼロセットされるという仮定の妥当性はいまだに検証されたことがない。地殻浅所にある断層は、測定試料の採取は容易であるが、断層運動により発生する摩擦発熱や地熱による影響が小さいため、高温になるとは考えにくい。そのため、地殻浅所での断層運動を想定した ESR 信号強度のゼロセットに関する研究が重要である。

石英中に見られる ESR 信号強度に対して破壊が及ぼす作用に関する先行研究の文献調査 (田中, 1987; 幡谷・田中, 1993; 福地, 2004) から、変位量と ESR 信号に関して次のようなことが指摘される。すなわち、(1) ESR 測定での測定条件が不適切であること、(2) 各 ESR 信号に対しての測定結果が明示されておらず、(3) グラフ化もされていないこと、(4) 摩擦発熱と ESR 信号の関係を考慮していないことである。そこで、本研究では、低封圧のためほとんど摩擦発熱しない地表付近での断層運動を想定し、珪砂試料 (JIS 試験用粉体) の剪断試験を行い、変位量と ESR 信号強度の関係を明らかにした。試料は内径 20 mm、外径 30 mm、厚さが約 1.5 mm の円筒状で、実験は東北大学に既設の低速リング剪断試験機を用いて、すべり速度、軸応力をそれぞれ 0.72 mm/s、0.981 MPa で一定にし、変位量は最大で 1.7 m である。

まず、断層運動に伴う発熱に関して、Sibson (1975) による式から剪断面での温度上昇 T を推定すると、本研究における条件では、数度の温度上昇に抑えられることがわかり、剪断試験によって発生する熱の影響は無視できると判断される。また ESR 測定に関しては、 $\equiv\text{Si}\cdot$ で表される (\cdot が孤立電子、 $-$ が共有電子対を示し、 \equiv は 3 つの Si 原子がその先で結合していることを示す) E_1' 中心が R 信号 (同様に石英中に見られるが構造は不明) と重ならないで検出される条件 (マイクロ波電力: 0.01 mW) やそのほかの信号も検出できる条件で行った。変位量と ESR 信号強度の関係から、 E_1' 中心、OHC (O^3)、Peroxy 中心 ($\equiv\text{Si-O-O}\cdot$) 等に関しては変位量の増加に伴う ESR 信号強度の減少が確認できた。この結果、地表付近の低封圧下においても、変位量がゼロセットに寄与することは十分に考えられ、浅所での断層運動がゼロセットを引き起こす可能性は十分に考えられる。

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キーワード：断層、電子スピン共鳴（ESR）、変位量、熱、温度上昇、ESR信号強度

Keywords: Fault, ESR: Electron Spin Resonance, Displacement, Heat, Temperature Rise, ESR Intensity